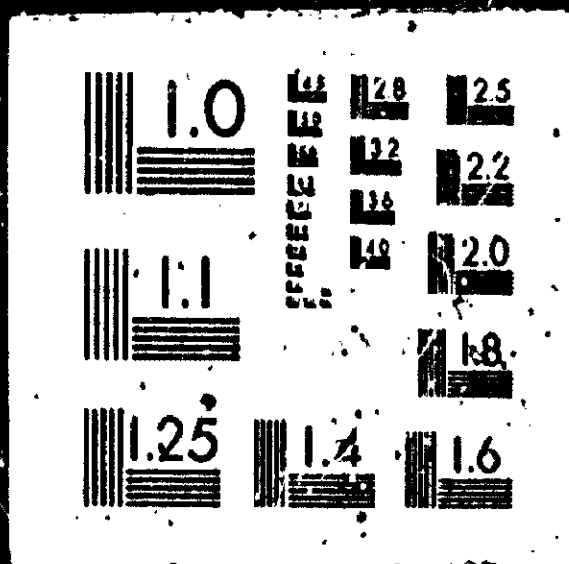


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Special Investigation Report
An Evaluation of the Garrett TPE 331 Engine's
Potential for Turbine Oil By-Product
Contamination of an Aircraft Cabin Environmental System

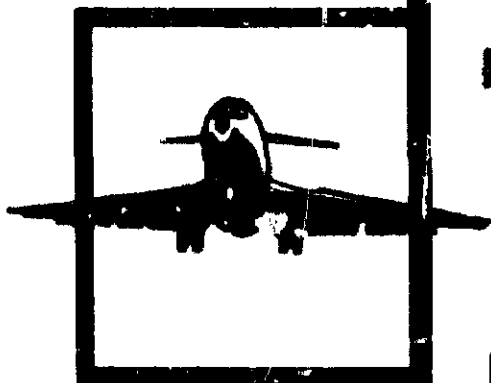
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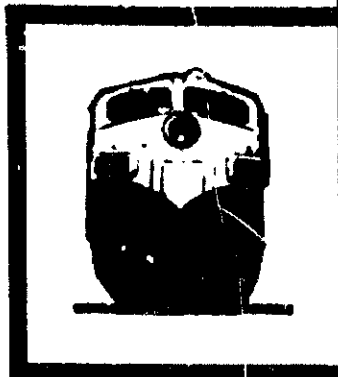


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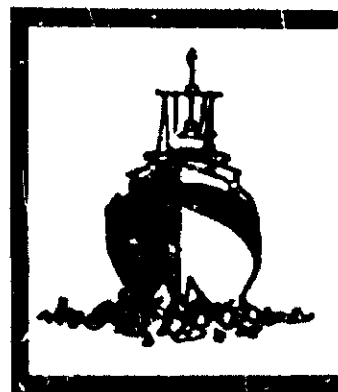


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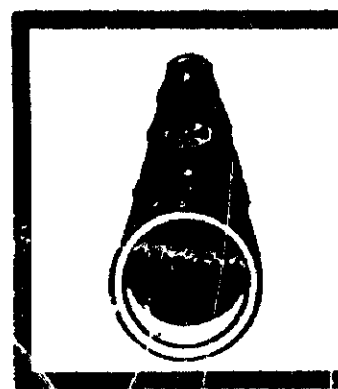
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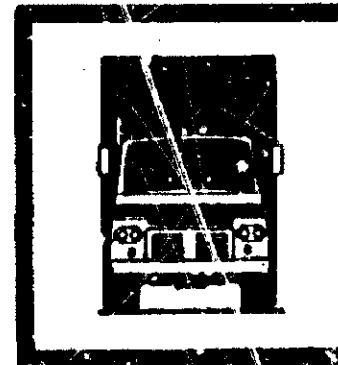
SPECIAL INVESTIGATION REPORT



**AN EVALUATION OF THE
GARRETT TPE 331 ENGINE'S
POTENTIAL FOR TURBINE
OIL BY-PRODUCT CONTAMINATION
OF AN AIRCRAFT CABIN
ENVIRONMENTAL SYSTEM**



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included the technical expertise for formulating the tests and the analysis of the test results. As a result of the investigation, the Safety Board has concluded that contamination of compressor bleed air by the ingestion of engine oil is not possible.

The National Transportation Safety Board approves this report for publication and distribution in the National Technical Information System in the interest of information exchange; however, the Safety Board's approval of the distribution of the report does not constitute a general endorsement or safety certification of the Garrett TPB 331 engine.

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**NATIONAL TRANSPORTATION SAFETY BOARD
WASHINGTON, D.C. 20594**

SPECIAL INVESTIGATION REPORT

Adopted: January 20, 1984

**AN EVALUATION OF THE GARRETT TPE 331 ENGINE'S
POTENTIAL FOR TURBINE OIL BY-PRODUCTS CONTAMINATION OF AN
AIRCRAFT CABIN ENVIRONMENTAL SYSTEM**

SYNOPSIS

Introduction

A hypothesis presented to the National Transportation Safety Board investigators during several accident investigations caused the Safety Board to examine the possibility that a cracked front mainshaft compressor carbon seal element used in the Garrett TPE-331 turboprop engine might allow engine oil to leak through the carbon seal, enter the engine's airflow, pass through the compressor bleed air systems, and allow toxic or anesthetic by-products of the oil to enter the aircraft's cabin environmental system. Further, that the toxic or anesthetic gases, if any, might adversely affect the flightcrew's capacity to control the aircraft during critical phases of flight. And finally, that the potential for a safety hazard could extend to all turbine engines using synthetic turbine oil and supplying compressor bleed air for aircraft cabin environmental control.

To test the validity of the hypothesis, the Safety Board formed an ad hoc Special Investigative Committee. The Committee received technical support from manufacturers, consultants, and other governmental agencies who aided the Safety Board investigators in investigating the hypothesis. These included the Federal Aviation Administration's Civil Aeromedical Institute, Clayton Environmental Consultants, Inc., the Department of Energy, the Environmental Protection Agency, Exxon Research and Engineering Company, and the Garrett Turbine Engine Company. The contributions of these organizations included the technical expertise for formulating the tests and the analysis of the test results. As a result of the investigation, the Safety Board has concluded that contamination of compressor bleed air by the ingestion of engine oil is not possible.

The results of a special investigation of the type undertaken by the Safety Board in this case are customarily reported in a Group Chairman's Report which is filed in the docket of the accident investigation(s). However, this special investigation involved an elaborate methodology which may be of general interest to the aviation community. Accordingly, the Safety Board has formally approved this report for publication and distribution in the National Technical Information System in order to promote information exchange.

Background and Test Protocol

The committee reviewed reports of tests and analyses of a range of similar engines and bleed air sampling techniques. As a point of departure, the design philosophy and operational configuration of the sealing around the main shaft of the Garrett TPE331 turboprop engine were studied and evaluated. The sealing in this and other similar engines

is designed to prevent, at all times, the engine lubricant from entering the compressor bleed-air path. Two seals are used in the TPE331 to prevent escape of the engine lubricant around the main shaft. First, a mechanical (carbon) seal is provided to prevent leakage if operating oil pressures exceed normal pressures and to prevent potential leakage that might occur during engine shutdown. The mechanical seal potentially is subject to wear and damage. Secondly, the TPE331 has a pressurized knife-edge labyrinth air seal that is specifically designed to prevent passage of air/oil mist from the reduction gearbox into the compressor chamber during engine operation. An important consideration in this design is that mechanical contact of the seal and shaft is not required to accomplish the pneumatic sealing.

In addition to the shaft seals, a negative air pressure differential of approximately 2 to 3 psig is maintained between the reduction gearbox pressure and the positive pressure in the compressor chamber; this design feature is intended to cause air to flow into the gearbox if the shaft seals fail, and the pneumatic sealing action would theoretically prevent the air/oil mist from escaping the gearbox into the compressor chamber.

The chemical composition of synthetic turbine oils also was reviewed and possible mechanical, chemical, and thermal breakdown products were identified. A certification test run on a large high by-pass ratio turbofan engine was of special interest. During this test, oil was injected into the compressor inlet while bleed-air samples were taken at the compressor bleed air discharge port. Carbon monoxide was the only contaminant that increased significantly; concentrations of unburned hydrocarbons and oil breakdown products remained low at compressor discharge temperatures up to 851° F. These results suggested that similar oil/air concentrations in the TPE331 would be expected to produce (1) undetectable or very low concentrations of unburned hydrocarbons and other contaminants, and (2) carbon monoxide well below permissible limits in the bleed-air.

Using available background information and the theories of sealing design in turbofan engines (particularly the TPE331), the committee developed a threefold testing protocol to answer the following questions: (1) Can oil leak from the engine oil system into the airflow, either during normal operation or following seal malfunction? (2) Would the characteristics of this leaked oil be changed in the engine compressor due to thermal degradation, volatilization, etc.? and (3) Would these breakdown products of the leaked oil be sufficiently toxic or anesthetic to affect crew performance?

The possibility of oil leakage was addressed in two series of tests -- one in which bleed-air was sampled from a production engine with a serviceable carbon seal, and the other involving an engine from which the carbon sealing element had been completely removed. The latter test was considered by the committee to be representative of the worst possible seal defect. To sample for oil breakdown products, heated oil was injected into the air intake of the operating engine and samples were taken of the bleed air discharge. The air samples were subsequently analyzed both qualitatively and quantitatively for potential contaminants. The potential for the breakdown products of the oil to debilitate a flight crew was assessed by analyzing the contaminants for toxicity, by considering the possible sources of contamination during the sampling, and by reviewing the evidence associated with nine identified candidate accidents and one incident.

Results and Conclusions

Results of the investigation indicated that the quality of bleed-air from the TPE-331 turboprop engine is not measurably different from the quality of ambient air. This is true for both a certified airworthy engine and an engine operating without a front

mainshaft compressor carbon seal element. During engine operation, the pneumatic main labyrinth shaft seal together with air pressure differentials provided by the design of the TPE331 effectively prohibited oil from escaping from the engine gearbox into the compressor airflow, regardless of the condition of the mainshaft carbon seal.

Analysis of bleed-air taken while Exxon 2380 turbine oil was injected into the engine's air intake indicated that the oil and its additives underwent no detectable change as they passed through the engine's compressor section. No toxic compounds of significant amounts were found in the bleed-air during any of the engine tests. (The TPE-331 is a relatively small, low pressure-ratio centrifugal compressor engine. Air in the compressor chamber is not subjected to temperatures of sufficient magnitude and duration for any significant degradation of oil (if present in the compressor air) to occur. Furthermore, the chemical composition of synthetic turbine oil is apparently not such as to produce toxic or anesthetic breakdown products at the temperature encountered in these tests.)

There was no evidence that toxic oil contamination of bleed-air could occur from damage or other malfunction of the front main shaft compressor carbon seal in the TPE331. Further, even with oil contamination of the bleed-air supply, oil breakdown products were not of sufficient concentrations to produce toxic or anesthetic effects on flight crew members.

As a result of the ad hoc Committee's investigative findings, it is concluded that the hypothesis concerning subtle pilot incapacitation due to engine oil contamination of the bleed-air supply from the TPE 331 engine entering the cabin aircraft's environment is completely without validity.

INTRODUCTION

Through a National Transportation Safety Board Investigator's safety proposal resulting from several accident investigations and reports of engine testing accomplished by a private consultant, Safety Board investigators became aware of a hypothesis with safety implications warranting a special investigation. The hypothesis concerned the Garrett TPE331 turboprop engine (See figure 1) and suggested that a cracked front main shaft compressor carbon seal element 1/ might allow engine oil to leak through the carbon seal and enter the engine's airflow path. The hypothesis further suggested that toxic or anesthetic gases might then be released from the compressor bleed air port into an aircraft cabin environmental system, and the gases might adversely affect a crew's capacity to control the aircraft during critical phases of flight.

1/ Two seals are used in the TPE331 engine for engine oil containment and to prevent the oil from leaking from the reduction gearbox into the engine inlet, passing into the compressor chamber, and potentially contaminating cabin bleed air. The first seal is the front main shaft compressor seal assembly. This type of seal is necessary to prevent oil leakage when the engine is not operating or is operating at higher than normal oil pressures. The second seal is a rotating labyrinth seal which is pneumatically pressurized to approximately 26 psi at the inner knife edges. (This type of seal has no sealing capability to prevent engine oil from exiting the reduction gearbox when the engine is not operating.)

Oil containment also is accomplished by pneumatic sealing consisting of a negative pressure of approximately 2 to 3 psi inside the reduction gearbox with respect to atmospheric conditions. A detailed discussion, including illustrations of the foregoing components used to accomplish oil containment is presented in appendix A. A discussion of the design philosophy, the operational configuration, and the potential failure modes is also included in appendix A.

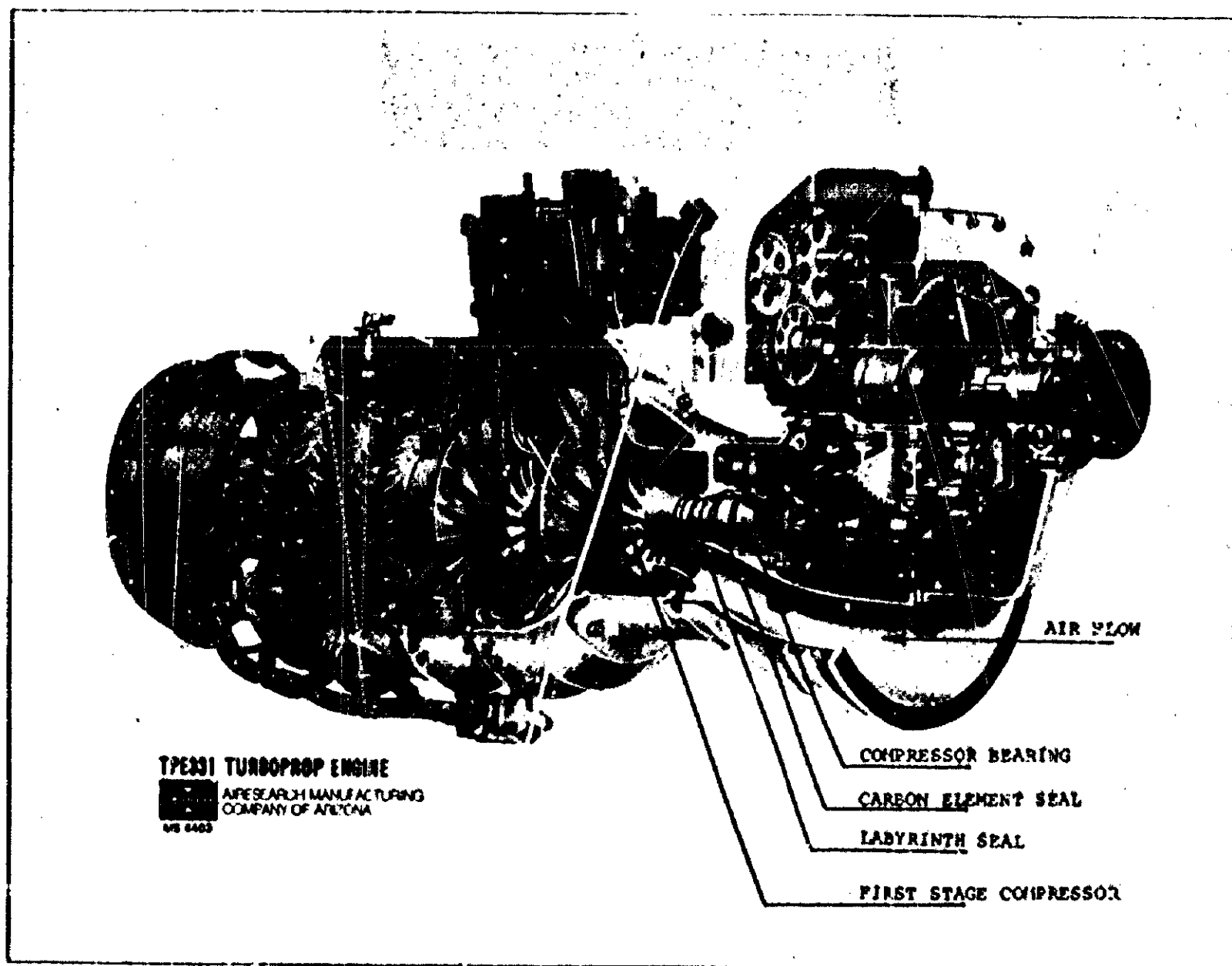


Figure 1.--TPE 331 turboprop engine.

In view of the potential applicability of the hypothesis to all turbine engines using synthetic turbine oil and supplying compressor bleed air for aircraft cabin environmental purposes, the Safety Board formed an ad hoc Special Investigative Committee. The committee received technical support from other governmental agencies and manufacturers whose expertise and facilities aided the Safety Board investigators in investigating the hypothesis.

The committee reviewed reports of similar tests and analyses that had been conducted on other engines in order to gain as much background information as possible on aircraft bleed-air contamination. The committee reviewed the chemical composition of synthetic turbine oils to determine their general chemical composition and to identify any potential breakdown products of synthetic turbine oils that might occur during operation of the engine. Additionally, the committee conducted two series of tests on a Garrett TPE331 engine to develop more complete data on the performance of the compressor seal and the effects of the engine compressor's operating environment on synthetic turbine oil. These tests were specifically designed to investigate the potential for oil leakage through a cracked front mainshaft compressor carbon seal and the possible release of toxic or anesthetic gases into the compressor bleed-air from oil entering the compressor chamber. Finally, reports of eight fatal accidents and one nonfatal accident were reviewed to determine whether or not these accidents could have been caused by incapacitation of the pilots by toxic by-products of turbine engine oil.

APPLICABLE AIRCRAFT ENVIRONMENTAL SYSTEMS

The results of this test program are applicable directly only to aircraft that use Garrett TPE331 engine compressor bleed air for their cabin environmental system(s).

DISCUSSION OF POTENTIALLY RELATED AIRPLANE ACCIDENTS

Nine accidents and one incident involving the possibility of pilot incapacitation occurred from 1979 through 1981 which resulted in the destruction of nine airplanes and a total of 38 fatalities. These accidents involved Mitsubishi MU-2 airplanes and four other models of airplanes all of which were equipped with Garrett TPE331 turboprop engines. The circumstances of these accidents were similar and were, therefore, reviewed to determine if subtle pilot incapacitation from oil fumes entering the airplane's cabin environment might have been a factor in these accidents. Eight of the accidents occurred on or near the final approach, and some involved abnormal or erratic movements of the airplane. Several of these accidents occurred in marginal weather conditions, such as rain, snow, fog, and darkness. Only one accident involved an engine failure; the airplane burned as a result of the accident. In one accident, the airplane continued to climb and repeated attempts to contact the pilot were unsuccessful; the airplane crashed into the Atlantic Ocean and was not recovered.

The accidents and incident are identified in table 1. Since the results of this special investigation indicated that oil fumes from a leaking carbon seal in a Garrett TPE331 engine could not enter the aircraft's cabin environment, the possibility of a cracked/broken compressor front main shaft carbon seal was dismissed as a factor in the accidents.

Table 1.--Accidents investigated for pilot incapacitation.

<u>No.</u>	<u>Date</u>	<u>Location</u>	<u>Airplane</u>	<u>Registration No.</u>
1	8/3/79	Hays, KS	Mitsubishi MU-2B	N208MA
2	11/1/79	Nashville, TN	Mitsubishi MU-2F	N8730
3	12/21/79	Provo, UT	Mitsubishi MU-2B	N2-OBR
4	1/11/80	Atlantic Ocean	Cessna 441	N441NC
5	2/14/80	Near Houston, TX	Mitsubishi MU-2B	N348MA
6	2/23/80	New Orleans, LA	Mitsubishi MU-2B	N962MA
7	12/8/80	Ramsey, MN	Mitsubishi MU-2Q	N969MA
8	12/15/80	Richmond, IN	Mitsubishi MU-2B-30	N93UM
9	1/7/81	Burns, OR	Aero Commander	N81521
10	4/22/81	Alpena, MI	Mitsubishi MU-2	N9JS

BACKGROUND AND TEST PROTOCOL

Background

Background Survey - Other Engines and Testing.--A review of engine and airframe experience was made to identify any previous testing that had been conducted on oil decomposition in turbine engines and the types of bleed air sampling techniques employed. Of special interest was a certification test that was conducted by a major engine manufacturer on a large turbofan engine used in wide-bodied air carrier airplanes. In that test, oil was injected into the compressor inlet while bleed air samples were taken at the

compressor discharge bleed port. The bleed air samples were analyzed by a laboratory utilizing a gas chromatograph (GC)/mass spectrometer (GC/MS). 2/

A number of bleed air contaminants were tested for carbon monoxide (CO), unburned hydrocarbons ($C_2 - C_6$), and oil breakdown products. During the testing, an oil breakdown product level greater than 0.5 parts per million (ppm) was never detected. The maximum level for unburned hydrocarbons was two (2) ppm. The only significant contaminant increase noted was CO.

Figure 2 shows the relationship between bleed air temperature at the turbofan engine's 16th stage compressor bleed port and the detected CO level obtained when oil was injected at a rate of 12 gallons per hour. Below approximately 600° F, no CO concentrations above ambient were noted. At 851° F, 49 parts per million (ppm) of CO were present. The CO levels varied as a function of the compressor discharge temperature. However, even with a 12-gallon per hour oil injection rate during the certification test, the maximum CO level of 49 ppm was below the 50 ppm prescribed limit of the Federal Aviation Administration (FAA) (14 CFR 25.831(b)(1) for passenger and crew compartment and the Occupational Safety and Health Administration (OSHA) limit for a 40-hour work week based on an 8-hour daily exposure. The FAA's permissible upper limit for CO level occurred at a compressor discharge temperature approximately 170° F higher than the 680° F (standard day) compressor discharge temperature of the TPE331.

Considering these data, and the 680° F compressor discharge temperature in the TPE331, oil/air concentration in the TPE331 at the same level as the tested engine might be expected to produce no detectable unburned hydrocarbons, no detectable oil breakdown products, and approximately 19 ppm CO in the bleed air. Nevertheless, it was deemed necessary to conduct similar testing on the TPE331 main engine to determine: (1) if oil would leak into the engine air flow through a compressor front shaft seal with a cracked carbon element and (2) with a known amount of oil injected into the air intake, what, if any, measureable oil and oil breakdown products would appear in the bleed air discharge from the compressor.

Description of Lubricating Oil Used in the Test Protocol.--Exxon Turbo Oil 2380 (ETO 2380) is an ester based synthetic turbo oil. Esters are synthesized from a family of alcohols known as polyols and low molecular weight fatty acids. In addition to the esters, a finished lubricant will contain antioxidants, additives to prevent corrosion and tricresylphosphate which serves as an antiwear agent. This brand of oil was chosen for these tests since this oil is the most widely used turbo oil in small general aviation turbine

2/ Gas chromatography (GC) is an instrumental method of analysis for the separation, identification, and quantification of the elements of volatile mixtures. "Permanent" gases (such as oxygen and carbon dioxide), volatile liquids, and pyrolyzed (chemical decomposition by heat) solids can all be separated by gas chromatographic techniques. The principles of gas chromatographic methods of analyses are discussed in numerous books and articles published on the subject. A basic description of the technique appears in a book by Walker, Jackson and Maynard entitled, "Chromatographic Systems - Maintenance and Troubleshooting," Academic Press Inc., New York (1977).

The major functions of a mass spectrometer M/S include (1) ionization of the molecules in the sample, (2) separation of the ions according to their mass to charge ratio (m/z), and (3) measurement of the relative abundance of each ion species for a given m/z value. The sample is introduced into an ionizing chamber. The ion fragments are accelerated and separated in a mass analyzer. The fragments then separate into their mass molecular weights according to their abundance.

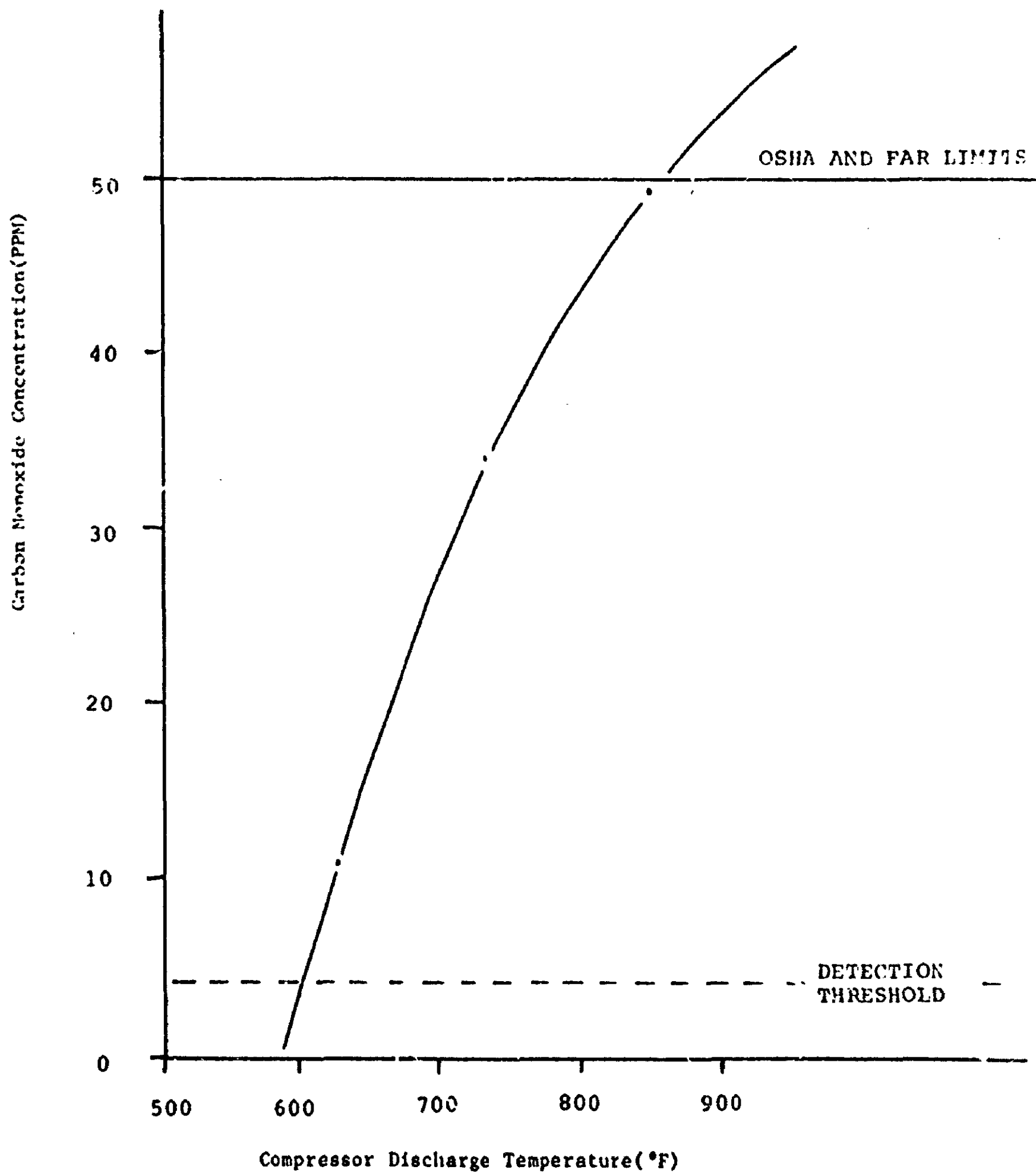


Figure 2.--Carbon monoxide concentration vs. compressor discharge temperature for a large turbofan engine.

engines, and the oil is typically representative of all turbo oils that are certified to conform to the test requirements of Military Specification MIL-L-23699C. A more detailed discussion of this oil is provided in appendix B.

Test Engine Description.--The TPE331-10-501M is a single shaft, turboprop engine, capable of developing 1050 shaft horsepower. In flight, the engine operates at a single rotational speed with the propeller rotating at 1,591 rpm. The main shaft of the engine, on which the compressors and turbine are mounted, rotates at a constant speed of 41,730 rpm. Normal bleed air pressures and temperatures are approximately 157 psi and 880° F at the above conditions. The normal engine airflow is 7.8 pounds per second (lbs/sec.)

Test Protocol

Test Organization.--In order to determine if the hypothesis presented in the introduction of this report was valid, a basic test protocol was developed at a coordination meeting that was held at the Garrett Turbine Engine Company, Phoenix, Arizona, on June 11 and 12, 1981. Participants at this meeting included the Safety Board ad hoc Special Investigative Committee and representatives of the Civil FAA's Aeromedical Institute (CAMI), the Department of Energy (DOE), the Garrett Turbine Engine Company, Mitsubishi Aircraft International, Inc., and Exxon Company, U.S.A.

The test protocol was conducted at the Garrett Turbine Engine Company during the week of July 20, 1981. Participants at the test protocol included representatives from the Safety Board's ad hoc Special Investigative Committee, CAMI, DOE, the Environmental Protection Agency, (EPA) Western Region, the Garrett Turbine Engine Company, Clayton Environmental Consultants, Inc., Mitsubishi Aircraft International, Inc., Exxon Company, U.S.A., and Exxon Research and Engineering Company. Observers to the test protocol included representatives from the Cessna Aircraft Company, Rexnord Incorporated, and American Aviation Services Corporation. Since the test protocol was designed to obtain data for toxicological evaluation, the CAMI representative was requested to function as the test coordinator to insure that the test data would be suitable for toxicological evaluation.

Development of the test protocol and analysis of the data obtained involved experts in the sciences and specialties of analytical and organic chemistry, chemical and mechanical instrumentation, mechanical engineering, industrial hygiene and medicine, and toxicology. Since the equipment needed by the experts to analyze the data was highly varied, it was necessary that the bleed-air samples be taken by the participating parties to their respective laboratories, where the samples were analyzed and the data interpreted and reported. The reported data were cross-reviewed by the Group Chairman and the other appropriate members of the groups that participated in the investigation. Additional independent verification of these data were provided by the CAMI and the DOE representatives.

Test Rationale - TPE331 Engine.--The rationale of the test protocol was to investigate the hypothesis that toxic or anesthetic gases could be generated from engine oil that leaked into the engine airflow through a broken seal and that these gases might adversely affect the crew's capacity to control the aircraft during critical phases of flight. For this hypothesis to be valid, two separate and distinct conditions would have to occur:

1. Oil would have to leak from the engine oil system into the engine airflow.
2. Oil which entered the engine airflow would have to undergo some change due to the conditions in the engine and would have to enter the compressor bleed air system. Further, the oil or the decomposition products would have to be toxic or anesthetic and present in sufficient quantities to adversely affect crew performance.

The test and analysis program was developed to answer the following questions:

1. If the carbon element compressor seal in the TPE331 engine is damaged, will the oil in the reduction gearbox leak through the seal, enter the engine's airflow, and be discharged from the compressor bleed air port?
2. If oil enters the engine compressor section, would any changes occur to its characteristics by volatilization, thermal degradation, oxidation, or any other process?
3. If the conditions in the engine compressor section are such that changes can occur in the oil, will any of the material produced have either a toxic or anesthetic effect on the occupants of the aircraft cabin?

To determine the answers to the above questions, the tests were designed and were divided into two major series--the first involved use of a production engine with a serviceable carbon seal, and the second involved use of an engine from which the carbon sealing element had been completely removed. The latter test was considered by the committee to represent the worst possible seal defect. A further test was undertaken during the first series in which heated oil, identical to that used in the engine, was injected into the air intake of the operating engine. This testing was undertaken to study the effects of oil injection on bleed air quality.

Consideration was given to use of an isokinetic sampling ^{3/} technique; however, since the vapors and gases would not undergo stratification, and because synthetic aviation lubricants are ash-free, it was expected that particulate matter would not form. While isokinetic flow rates may have been achieved during these tests, no specific effort was made to maintain isokinetic conditions, since these conditions were not deemed necessary. A more detailed discussion of this rationale is in appendix C.

Cracked Seal Test Configuration Evaluation.--During the coordination meeting of June 11 and 12, 1981, an evaluation was made to determine the optimum seal configuration to be used during the second bleed air test. The objective was to simulate the seal condition which was most likely to result in an oil leak past the seal. Since the test objective was to determine what occurs when a carbon seal cracks, a suggestion was made that a cracked seal be simulated by cutting the carbon element of an intact seal.

^{3/} In order to achieve isokinetic sampling, it is necessary that the mass flow within the stream being sampled be the same as the flow rate within the sampling probe.

A review was made to determine the width of a cut that would most likely result in oil leakage past the seal. The evaluation indicated that with a cut seal, the air path into the reduction gearbox would remain partially restricted, and the oil scavenge pumps would be able to maintain some negative pressure in the reduction gearbox. Thus, the narrower the cut, the higher the pressure differential across the seal, which would result in a higher airflow velocity through the crack in the seal. Finally, the higher the airflow velocity, the more difficult it would be for oil to pass through the crack. Thus, it was determined that the condition most likely to result in oil passing the seal was the condition with the carbon sealing element completely removed. Removal of the seal element would result in the lowest differential pressure across the seal, which would result in the lowest airflow velocity through the seal, as well as offer the maximum area for potential oil passage.

The following question was also evaluated: Would complete removal of the seal's carbon element eliminate any potential for seal/rotor contact at the edge of the crack to mechanically cause or allow oil to pass through the seal?

As a result of this inquiry, it was determined that since the cracked carbon element was stationary and the rotor was turning, no mechanical oil transfer (oil pumping) action would occur. It was determined that this action was further prevented by the centrifugal (slinger) action of the rotor face. It was, therefore, decided that the seal configuration with the carbon element completely removed was the one most likely to result in oil leakage past the seal. Consequently, this configuration was the one selected for testing. Figure 3 shows a comparison of the differences in configuration between a normal seal assembly and a seal assembly with the carbon element removed.

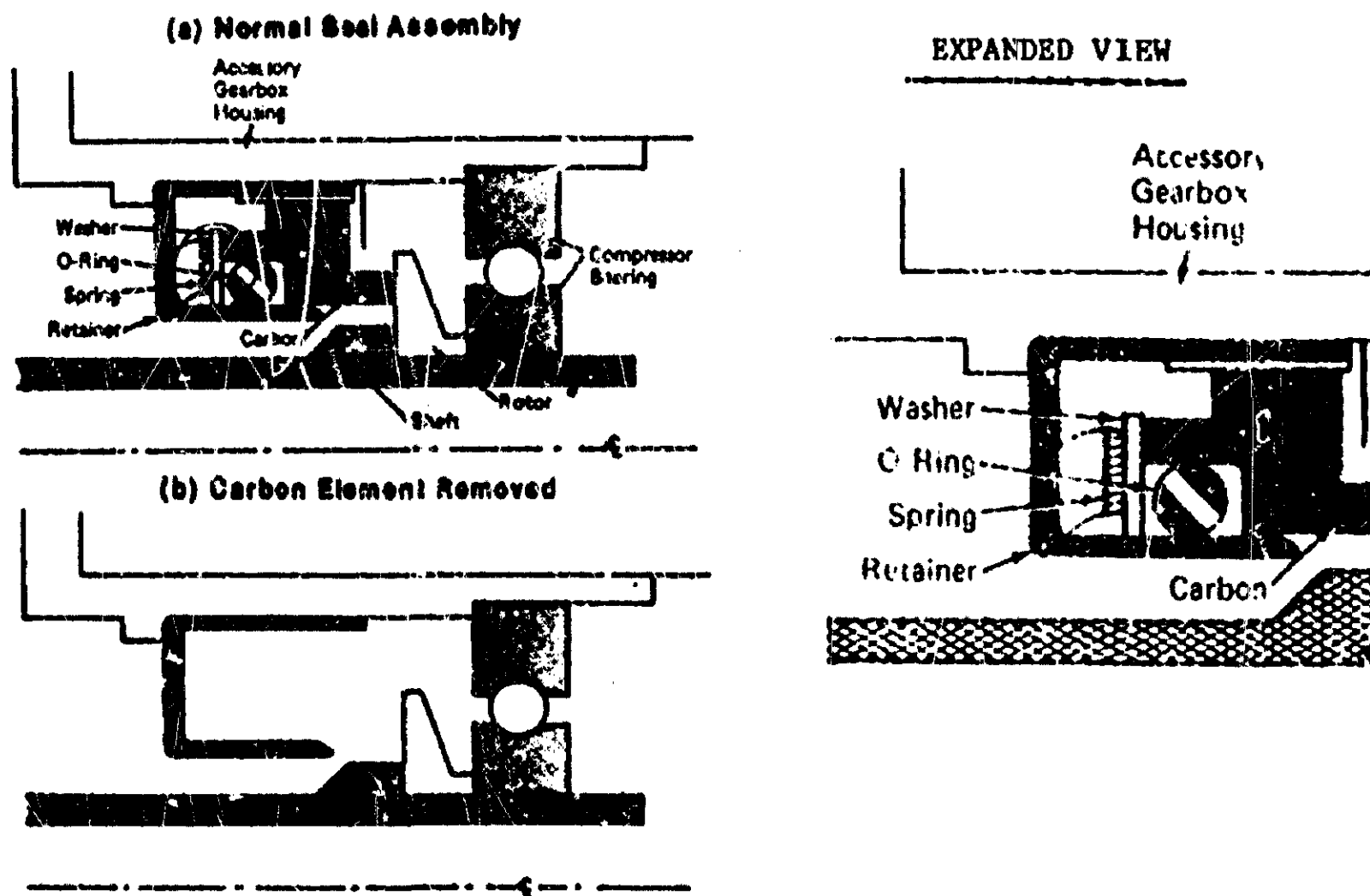


Figure 3.--Comparison of seal configurations.

Test Engine Configuration.--A certified production configuration TPE331-10-501M engine, serial number P36340, shown in figure 4, was drawn from the Garrett engine stock for the testing. No modifications were made to the engine for the first test series. For the second test series the engine's front main shaft compressor carbon seal element and associated internal components were removed. The engine was installed in an outdoor test stand that was located about 300 feet from an active runway. Hartzell HC-B4TN-5GL propeller was installed on the engine. The engine and the bleed air test apparatus are shown in figure 5. One percent of the engine's normal airflow of 7.2 lbs/sec. was bled from the compressor bleed port for the bleed air contamination tests.

Oil Injection Test Setup.--For the purpose of the oil injection test, an oil injection probe was mounted on the engine inlet (See figure 4) to allow injection of oil directly into the engine airflow. The tip of the probe was positioned so that the injected oil would impinge upon the hub of the first stage compressor impeller. This position was selected to simulate leakage at the compressor bearing. This is the only possible oil entry area into the compressor bleed air flow path, assuming that the structural integrity of the reduction gearbox has not been compromised.

The oil for injection was retained in a heated oil supply tank and was pumped to the injection probe through a series of valves and calibrated orifices that allowed control of the oil flow rate. The oil was heated to its normal operating temperature in the accessory gearbox.

Engine Installed Bleed Air Sampling Equipment.--A normal aircraft (Mitsubishi) bleed air flow control sonic nozzle was used to extract the bleed air from the aircraft bleed air tube. (See figure 5.) The nozzle was calibrated to provide a cross-check on the amount of air that was bled. The airframe anti-ice port was capped since this port was not used during the test. A detailed description of the system is presented in appendix D.

Condensed Review of Test Series.--The air sampling measurements were accomplished by Garrett Turbine Engine Company, Clayton Environmental Consultants, Inc., who provided technical consultation to Garrett Turbine Engine Company, Exxon Research and Engineering Company, and the Environmental Protection Agency.

The test program consisted of conducting two engine test series with appropriate instrumentation. In all of the tests, the engine was operated at a rotor speed of 41,730 rpm; engine operational parameters and test data were obtained and recorded. (See table II.) A tabulation of the various sampling techniques that were employed by the Garrett Turbine Engine Company, and Engineering Company, Clayton Environmental Consultants, Inc., Exxon Research and Engineering Company, and the Environmental Protection Agency, for each of the test data points for the two test series are presented in table III.

The Series 1 tests were conducted on July 21, 1981, and were designed to investigate the effect(s) of oil ingestion on bleed air quality. Following preliminary tests of engine intake air and bleed air from a normal engine, bleed air was sampled while injecting ETO 2380 turbo oil directly into the engine inlet. Oil flow rates ranging from 2 to 12 pounds per hour (lbs/hr) were used, followed by a separate test using a 12 lbs/hr injection rate, which represents the rate that would consume the entire engine oil supply in 1 hour. The oil flow rate of 12 lbs/hr gave an oil/air concentration 2.6 times greater than that used in the earlier discussed background survey turbofan tests where 12 gallons/hour (96 lbs/hr) were injected into the compressor. The bleed air was sampled continuously throughout the injection period and very sensitive instruments were used to search for bleed air contaminants.

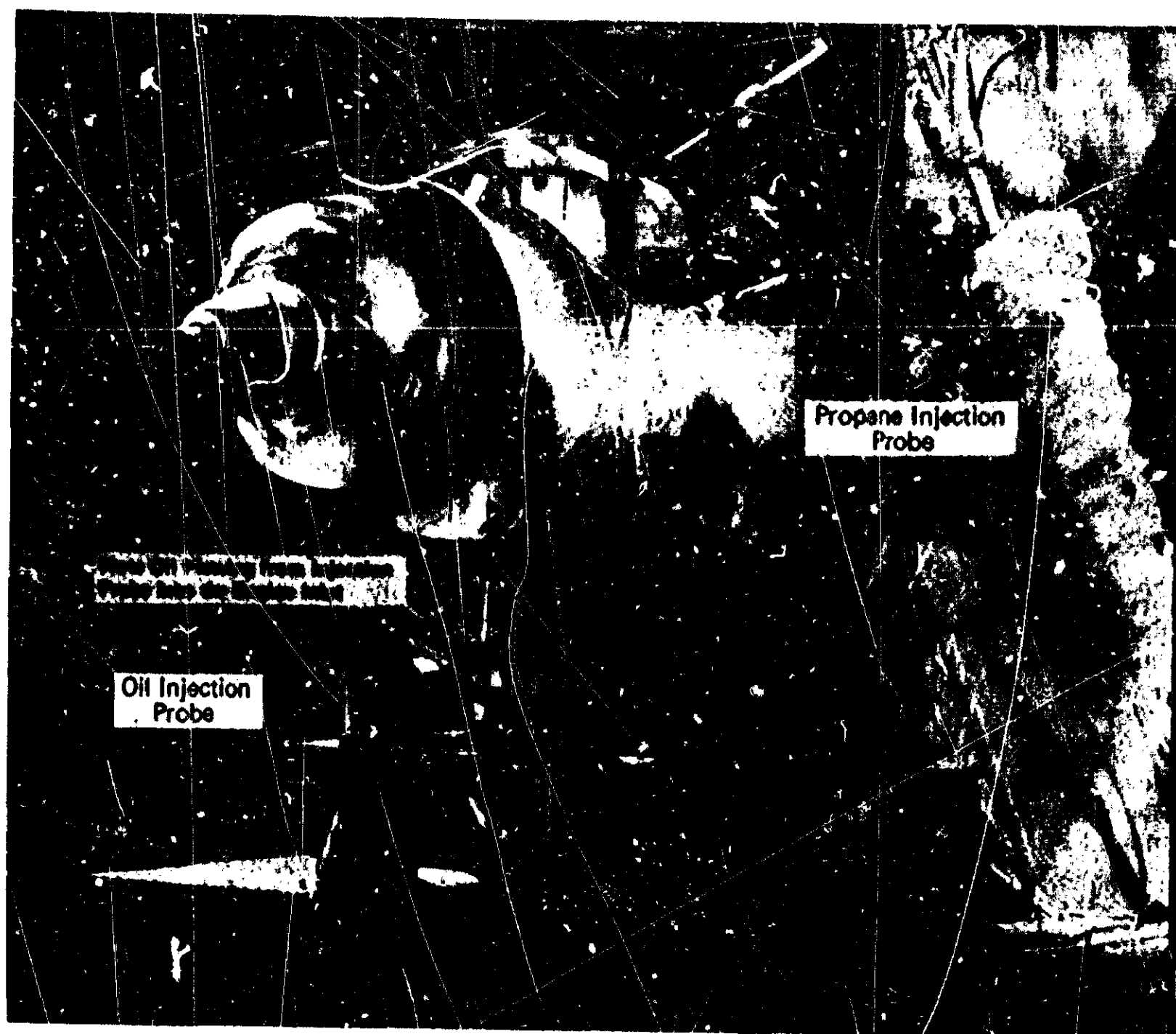


Figure 4.--Engine test configuration.

Series 2 tests were conducted on July 22, 1981, and were designed to investigate the possibility that engine oil might pass through a cracked front main shaft compressor seal carbon element and then enter the engine airflow. In these tests, the entire carbon sealing element was removed so that the worst condition to promote oil leakage might be investigated. The engine was run for 1 1/2 hours while the bleed air was sampled as in Series 1 tests.

Since the sampling and analytical methods employed in the foregoing tests were chosen to realize maximum sensitivity in measuring for airborne contaminants, the quality of the ambient air ingested by the turboprop engine had to be considered. For this reason, samples of the ambient air upwind of the engine's intake were taken using the same methods as for bleed air sampling. Throughout all the tests, Clayton sampled the ambient air at the test cell's fence line (fence line ambient) approximately 20 feet in front of the engine. These tests were made to determine the nature and quantity of contaminants being introduced in the ambient air thereby allowing for their exclusion from any



Figure 5.--Engine and bleed air test installation.

Table II.--Engine test data operational parameters and identification data.

Date of Test		7/21/61				7/21/61					
Engine Configuration		Standard (Good) Seal No Oil Injection				Standard (Good) Seal - No Oil Injection					
Engine Test Designation		Background Sampling - 1 Hour				Compressor Bleed Air Sampling - No Oil Injection					
Test No.		1				2					
Test Parameter	Units										
Time of Day		11:15	11:30	11:45	12:00	12:55	13:10	13:25	13:40	13:55	14:10
Date Point		01	02	03	04	05	06	07	08	09	010
Barometric Press.	In. Hg	29.75	29.73	29.73	29.73	29.68	29.68	29.63	29.65	29.65	29.64
Compressor Inlet Total Temp. (T ₂)	°F	102	104	103	103	106	106	107	109	109	107
Compressor Disch. Total Temp. (T ₃)	°F	715	715	717	716	719	720	721	717	717	718
Exhaust Gas Total Temp. (T ₅)	°F	1020	1027	1028	1029	1024	1025	1018	1019	1022	1023
Torque Diff. Press. Delta P	psi	40.8	40.5	40.5	40.5	40.5	40.5	40.5	40.5	40.5	40.5
Torque	In. lbs	24100	24000	24000	24100	24000	24000	24000	24000	24000	24000
Fuel Flow W _f	lbs. Hr.	394	393	393	393	394	394	393	391	390	391
Vibration (Gearbox)		.74	.78	.81	.83	.81	.80	.85	.87	.80	.78
Vibration (Turbine)		.23	.24	.26	.26	.24	.26	.27	.26	.24	.23
Compressor Disch. Total Press. (P ₀₃)	In. Hg	210	210	217	217	215	214	214	213	214	214
Compressor Disch. Static Press. (P ₀₄)	In. Hg	217	218	218	216	215	214	213	213	214	213
Engine Oil Press.	psi	101	101	101	101	101	101	101	101	91	101
Engine Oil Temp.	°F	163	164	164	164	164	164	164	164	164	164

Date of Test		7/21/61						7/21/61	
Engine Configuration		Standard (Good) Seal With Oil Injection						Standard (Good) Seal With Oil Injection	
Engine Test Designation		Compressor Bleed Air Sampling With Millipore Filter						Compressor Bleed Air Sampling for 15 minutes Engine shutdown to remove in-line glass cool filter	
Test No.		3a	3b	3c	3d	3e	3f	3g	
Test Parameter	Units								
Time of Day		15:40	15:50	15:55	16:00	16:08	16:15	16:45	
Date Point		011	012	013	014	015	016	017	
Rate of Oil Inq.	lbs/Hr	3	4	8	8	15	15	12	
Barometric Press.	In. Hg	20.63	20.63	20.63	20.63	20.63	20.63	20.59	
Compressor Inlet Total Temp. (T ₂)	°F	110	110	110	111	111	112	110	
Compressor Disch. Total Temp. (T ₃)	°F	721	721	724	724	724	721	726	
Exhaust Gas Total Temp. (T ₅)	°F	1039	1042	1040	1038	1050	1055	1065	
Torque Diff. Press. Delta P	psi	40.5	41.0	40.5	40.5	40.9	41.4	40.0	
Torque	In. lbs	24000	24200	24000	24000	24200	24200	24250	
Fuel Flow W _f	lbs/Hr	394	394	393	393	392	390	393	
Vibration (Gearbox)		.84	.83	.85	.86	.83	.86	.93	
Vibration (Turbine)		.23	.24	.23	.28	.24	.23	.25	
Compressor Disch. Total Press. (P ₀₃)	In. Hg	214	215	213	214	213.5	213	211.5	
Compressor Disch. Static Press. (P ₀₄)	In. Hg	213	214	213	214	214	214	212	
Engine Oil Press.	psi	101	101	101	101	101	101	101	
Engine Oil Temp.	°F	165	165	165	165	165	165	165	

Table II (con't).--Engine operational parameters and test identification data.

Date of Test		7/22/81						
Engine Configuration		Carbon Seal Removed No Oil Injection				Carbon Seal Removed No Oil Injection		
Engine Test Designation		Compressor Bleed Air Sampling 15 Hours				(5a) No Compressor Air Sampling and (5b) Start run with oil in night. Sampling immediate from start		
Comments		Engine shutdown for one hour. Collect and measure oil drip- pin, 1515 Inlet				Test 5a - Start up, 15 minute run to clear inlet. Engine shutdown to 1515, oil to drop into air inlet in preparation for 5a by start time.		
Test No.		4				5a and b		
Test Parameter	Units							
Time of Day		9:30	10:00	10:30	11:00	12:50	13:20	
Date Point		81	82	83	84	1516	1525	
Barometric Press.	In. Hg	28.70	28.70	28.70	28.70	28.68		
Compressor Inlet	°F	94	95	97	101	102		
Total Temp. (T ₂)	°F	704	707	710	709	714		
Compressor Disch.	°F							
Total Temp. (T ₁)	°F	998	998	1013	1013	1022		
Exhaust Gas	°F							
Total Temp. (T ₃)	°F							
Torque Dist. Press.	psi	-	-	-	-	-		
Delta P								
Torque	In lbs	-	-	-	-	-		
Fuel Flow W _f	lbs/hr	488	485	487	488	489		
Vibration (Gearbox)		.80	.75	.80	.83	.85		
Vibration (Engine)		.76	.75	.75	.74	.78		
Gear Case Pressure	In./100	+21	+23	+26.5	+27.1	+27		
Compressor Disch.	In. Hg	224	223.5	222.5	222.5	222.5		
Total Press PT ₁	In. Hg							
Compressor Disch.	In. Hg							
Static Press. (P ₁)	psi	102	102	101.5	101.5	101		
Engine Oil Press.	psi	101	102	102	102	101		
Engine Oil Temp.	°F							

NO ENGINE DATA RECORDED

NO ENGINE DATA RECORDED

Table III.--Tabulation of bleed air sampling techniques.

Test No.	COLLECTION SYSTEM						GAS ANALYSIS			
	Cryo- genics	Tenax	Char- Coal	Tenax and Char.	Bi- Sulphite	Milli- Pore Filter	CO (NDIR)	CO ₂ (NDIR)	HC (PID)	NOx (TECO)
1	E	C EPA	C	C	C	-	Q	Q	Q	Q
2	E	C EPA	C	C	C	-	Q	Q	Q	Q
3a	-	-	-	-	-	Q	Q	Q	Q	Q
3b	-	-	-	-	-	C	Q	Q	Q	Q
3c	-	-	-	-	-	C	Q	Q	Q	Q
3d	-	-	-	-	-	C	Q	Q	Q	Q
3e	-	-	-	-	-	C	Q	Q	Q	Q
3f	-	-	-	-	-	C	Q	Q	Q	Q
3g	E	C EPA	C	C	C	-	Q	Q	Q	Q
3h	E	C EPA	C	C	C	-	Q	Q	Q	Q
3i	-	-	-	-	-	-	-	-	-	-
3j	E	C EPA	C	C	C	-	Q	Q	Q	Q

E - EXXON
C - CLAYTON
Q - GARRETT

NDIR - Non-Dispersive Infrared
PID - Flame Ionization Detector
TECO - Chemiluminescent Analyzer

contamination found in the bleed air. Additionally, Clayton's samples were drawn from a glass manifold in a similar fashion as those of the engine bleed air. The results of analysis of these ambient air samples were compared with those from the fence line ambient samples to determine if the glass manifold testing system contributed or retained contaminants in the air reaching the sampling media.

Lubricating oil which may have undergone decomposition would be expected to exhibit physical and chemical properties different from the undecomposed oil. Samples of oil must taken during Test 3 of the Series 1 tests (oil injection tests) were collected to study the chemical composition of oil which had passed through the engine's compressor sections. Such samples would also provide an indication of the degree of bleed air contamination caused by additives, such as tricresylphosphate (TCP).

Bleed Air Sampling Methods and Procedures.--This section provides a condensed review of the sampling methods and procedures that were used for the test series. This section also contains a discussion and an evaluation of the test results. Figure 6 shows an overall view of the Exxon, BPA, and the Clayton bleed air sampling systems. Figures 7 and 8 are schematic representations of the bleed air sampling systems employed by Clayton Environmental Consultants, Inc., and Exxon Research and Engineering Company. A detailed discussion of the sampling systems and procedures that were employed is provided in appendix F. Additionally, a detailed discussion of the test results is contained in appendix F.

Garrett Turbine Engine Company's emission analytical equipment (mobile gas analysis unit) is permanently mounted in an automotive van. The van was positioned adjacent to the engine test cell. A stainless steel bleed air line extended from the bleed air manifold through a metal container containing a glass wool liquid/vapor separator filter, and into a heated line that led from the test cell into the van. Bleed air could be sampled continuously and analyzed in this system. A propane probe was mounted in front of the engine air inlet to provide a means of introducing propane, at will, into the inlet to test the response of the analytical instruments to a given surge of a hydrocarbon. Thus, the ability of the gaseous contaminants to pass freely through the glass wool liquid/vapor separator filter was demonstrated frequently during the testing by injecting propane directly into the engine inlet airflow which showed up immediately on the flame ionization detector 4/ (FID) reading. The injection of propane allowed verification of the FID system operation during periods of very low readings. This probe is shown in figure 4. The effect of liquid contamination on the sample lines is shown in test 3g when the glass wool liquid/vapor separator filter was removed and an oil injection rate was established. If the FID readout gave a positive reading with no change in the CO and carbon dioxide (CO_2) levels, it would demonstrate that the oil had passed through the engine and into the bleed air system with no change in composition.

Clayton used a series of collection methods. For the most part, ambient air and bleed air were passed into a manifold. Samples were collected from the manifold by adsorption onto adsorbent materials which consisted of Tenax, 5/ activated charcoal or

4/ A flame ionization detector is an electronic instrument in which the sample gas passes through a flame sustained by regulated flows of air and a premixed hydrogen/diluent fuel gas. Within the flame, complex ionization processes occur which are related to the presence of unburned hydrocarbons in the sample being analyzed.

5/ Tenax is a polymeric material that is widely used in environmental air sampling procedures. Contaminants in the sampled air are physically adsorbed on the surface of the adsorbent material and are then later desorbed for analysis.

EXXON's equipment is in the foreground followed by EPA and Clayton equipment. Garrett's equipment van is not visible and is located to the right of the visible equipment.

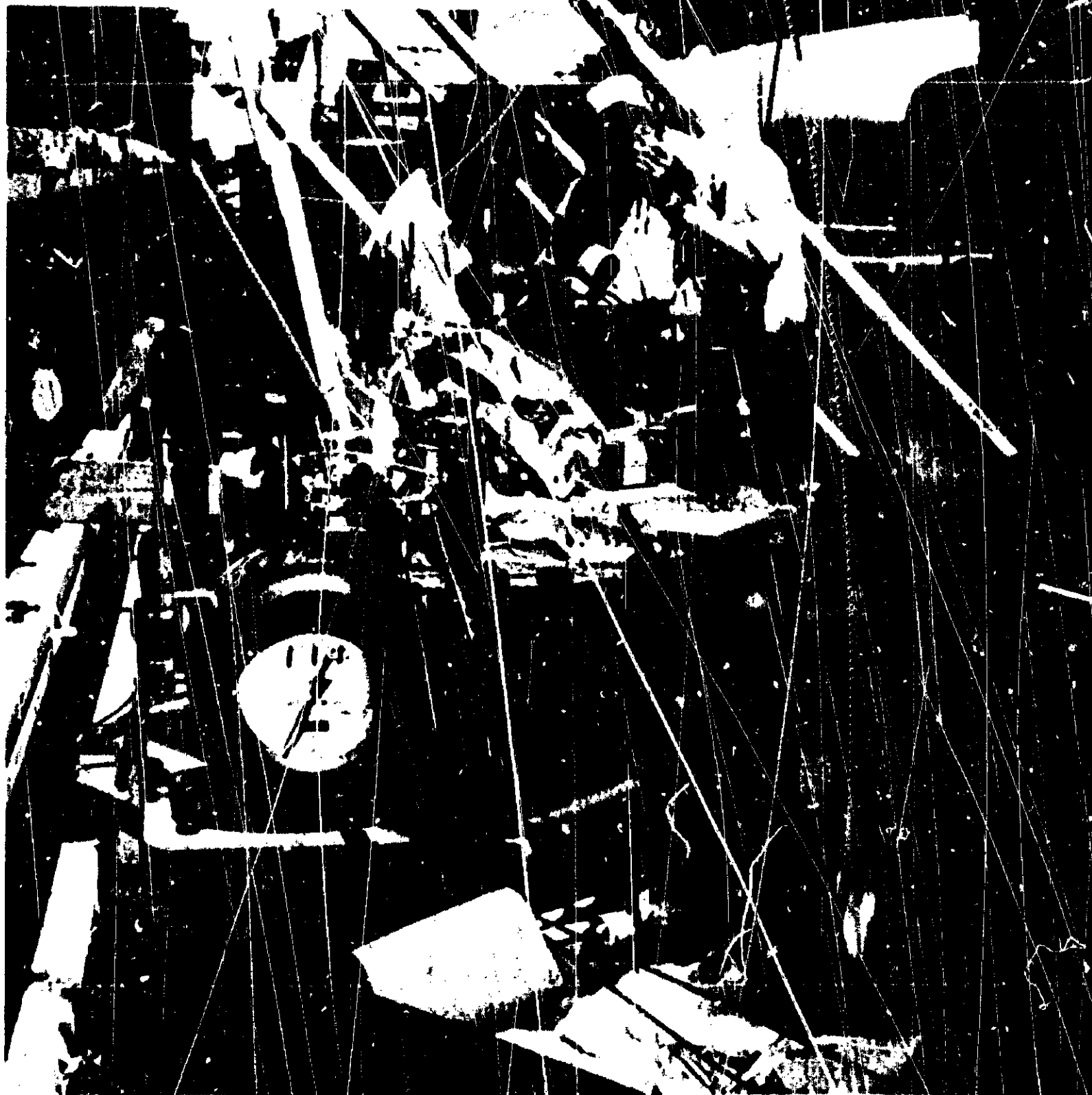


Figure 6.--Overall view of Exxon, EPA and Clayton bleed-air sampling equipment.

Tenax and activated charcoal packed in series in tubes. Sampling for aldehydes was carried out by bubbling the air through a sodium bisulfite solution. Ambient air was sampled primarily by use of adsorption in Tenax columns. For tests 3a, b, c, d, e, and f, bleed air was directed through preweighed membrane filters used for sampling oil mists.

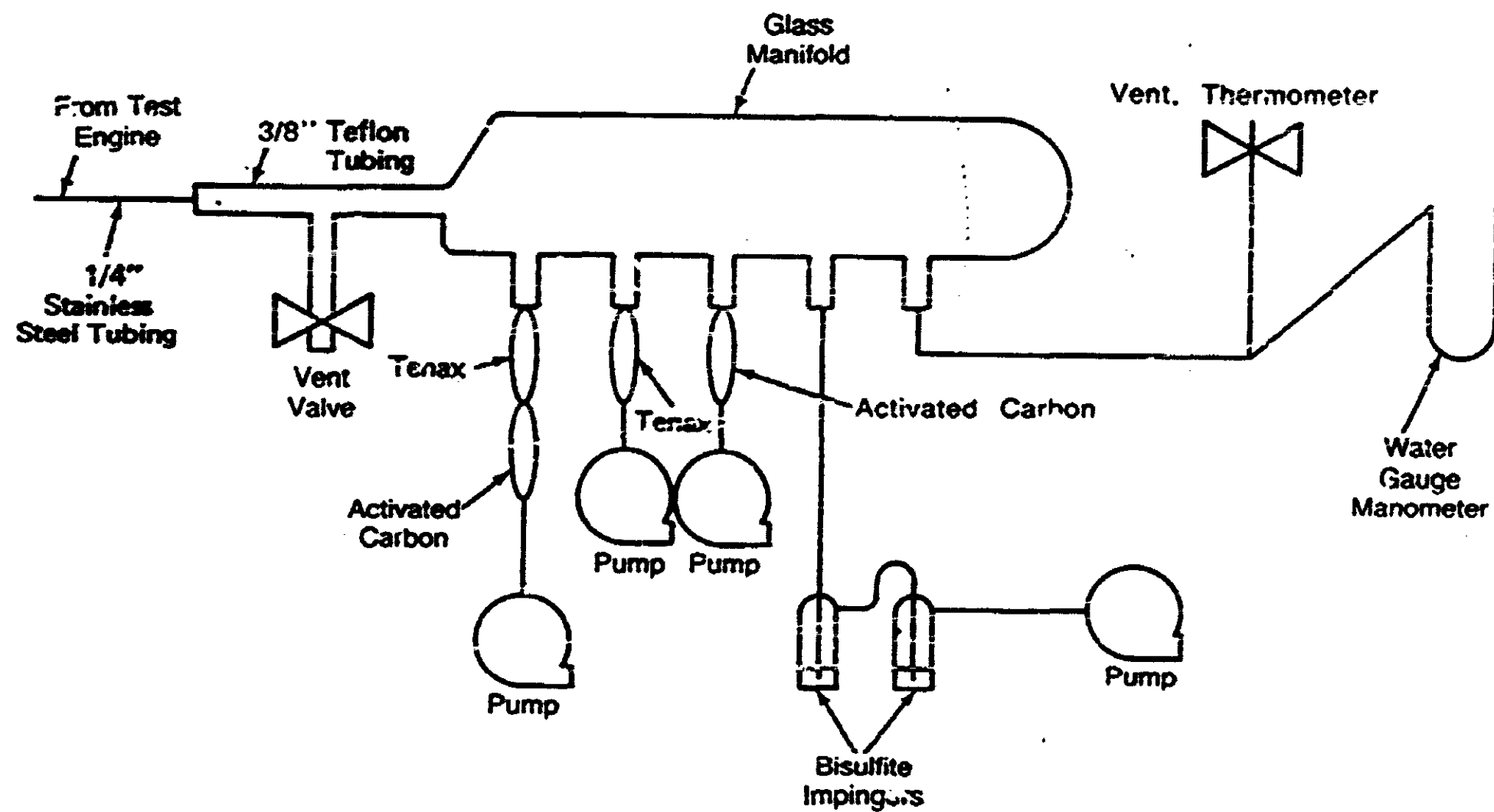


Figure 7.--Schematic of Clayton's inlet and bleed-air sampling system.

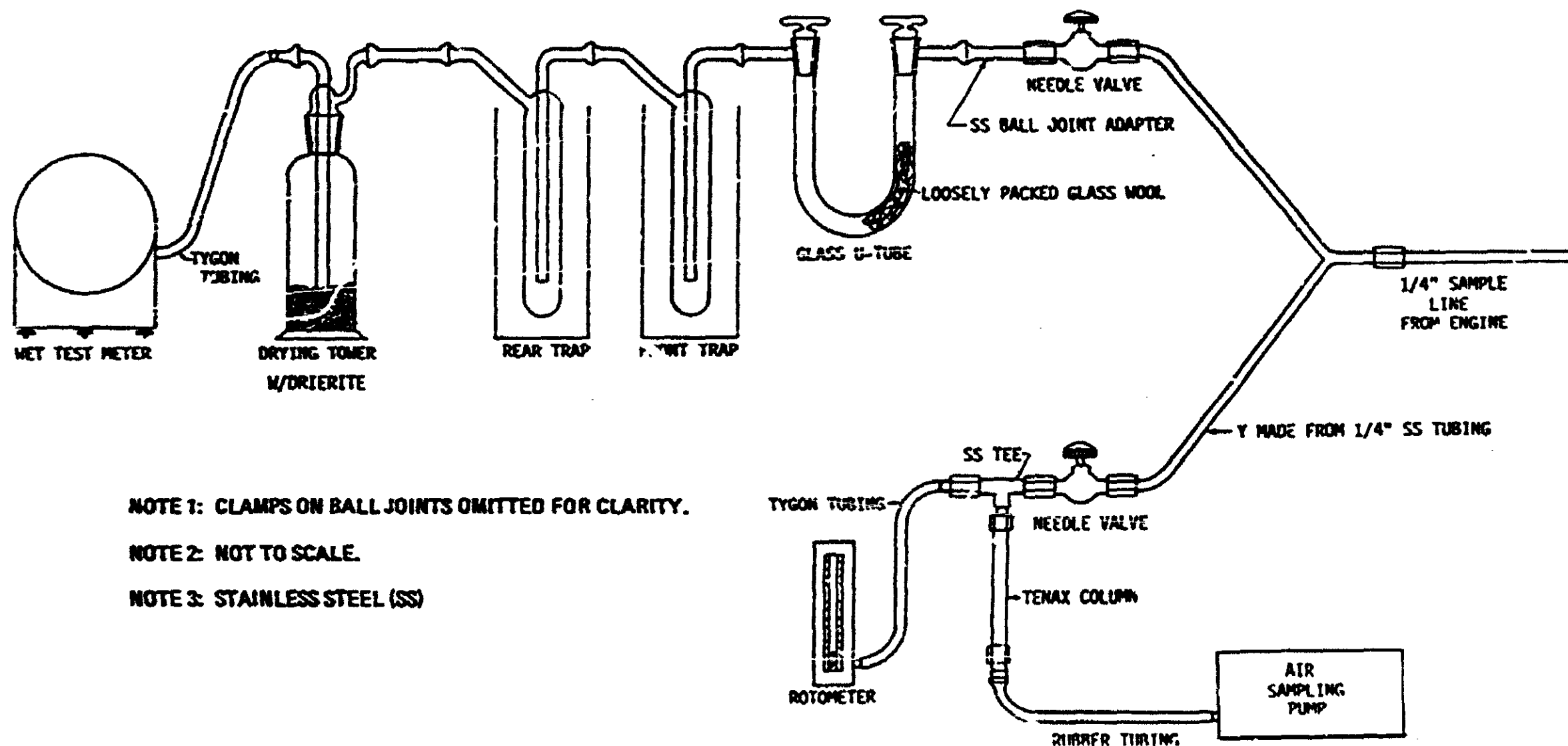


Figure 8.--Sampling train used by Exxon in Garrett TPE331 engine test.

Two methods of ambient and bleed air sample collecting were used by the Exxon Research and Engineering Company. The first method employed was cryogenic trapping which consisted of passing the bleed air stream through two glass traps cooled to -78°C (-108°F). This method has been used previously by Exxon to trap decomposition products of oil for analysis. The second method employed was to pass the air stream through a packed column of Tenax which removed contaminants from the air.

The ambient and bleed air that were sampled by the Environmental Protection Agency were passed at a controlled rate from the sample lines through tubes containing Tenax. Sampling was continuous throughout the prescribed sampling period of each test.

Conduct of Tests

Series 1 Tests.---The following tests were conducted on July 21, 1981:

Tests 1 and 2

Tests 1 and 2 were considered to be representative of ambient or background air conditions. These tests were conducted with the front main shaft compressor carbon seal element installed in the test engine.

Test 1 was designed to test ambient or background air for contaminants in the air entering the engine intake. No oil injection was performed during this test. While the engine was running, air was sampled for 60 minutes through a probe mounted on the test stand. After test 1 was completed, the engine was stopped and lines were changed so that compressor bleed air could be sampled. The engine was then started, operated a few minutes, and then compressor bleed air was sampled for 90 minutes. This test was designated as test 2 and represented compressor bleed air from a normally operating TPE331 with an intact seal.

Test 3

This was a compressor bleed air oil mist sampling test with a standard front main shaft compressor seal installed. This test was designed to simulate an engine operating with a sizeable oil leak into the first stage of the compressor. Lines from the previously discussed heated oil supply tank containing ETO-2380 turbo oil at 160°F led to an injection probe that was mounted so that the injected oil could be directed into the air inlet and would impinge directly upon the first stage compressor impeller. (See figure 5.)

The engine was started and operated to achieve a steady stall condition. Then graduated oil injections at 2, 4, 6, 8, 10, and 12 lbs/hr were made progressing from lowest to highest rates. While the engine was running, oil injection was begun, and after a few minutes of operation to achieve a steady state, bleed air sampling was carried out for a period of 5 minutes during each level of oil injection. After the graduated series of oil injections were made, the engine was stopped and the glass wool filter in Garrett's sampling lines was removed. The engine was restarted, run for several minutes to achieve a steady stall condition, and then oil was injected at a rate of 12 lbs/hr. After a few minutes of operation, bleed air sampling for airborne oil decomposition products was carried out for 15 minutes. This terminated the sampling in the Series 1 tests.

During the foregoing tests, Garrett's on-line analytical train was used to sample bleed air at all levels of oil injection. Clayton also sampled at all levels of injection by using a 37-millimeter cassetted in-line membrane type millipore filter system to obtain oil mist samples directly from the stainless steel sample transfer line. This line drew unfiltered bleed air from the engine-installed bleed air collector.

Series 2 Tests

Test 4

This test was designed to simulate engine operation with a defective carbon seal. Overnight, the engine was disassembled and the standard front mainshaft compressor seal assembly carbon element and the seal's internal components were removed from the engine and replaced with an empty seal retainer. The engine was reassembled in preparation for tests to simulate operation of an engine with a defective seal. On the morning of July 22, the bleed air lines were cleaned with Freon and the engine (after having been stopped to tighten housing bolts) was run 15 minutes with the sample lines open to remove any contamination that might have occurred during an overnight dust storm. Once the engine had been running for about 15 minutes, sampling was begun and continued for 90 minutes with the engine running continuously. The engine was then shut down to allow the residual oil to collect in the accessory gearbox, after the normal oil scavenging process had ceased. The oil that passed through the seal retainer was then collected and measured.

Test 5

This test was designed to simulate a "dirty" startup of an engine having a leaky or defective carbon seal which allowed oil to drip into the air inlet and onto the ground. It was accomplished in two steps, 5a and 5b. In both tests, the engine was operated in the same configuration as in test 4. No bleed air samples were obtained during test 5a. For Test 5a, the engine was started and operated for 10 minutes in order to clean the inlet. The engine was then shut down to allow oil to drip and accumulate into the air inlet, in preparation for a "dirty start" (Test 5b) test. Test 5(b) consisted of starting the engine with the oil accumulation in the inlet and collecting bleed air samples for 10 minutes immediately after the engine was started.

The engine was then shut down and the propeller unfeather pump was activated in order to demonstrate that an increase of oil drippage into the inlet as the oil level rose occurred. This was accomplished to assure that no metal to metal contact existed between the seal retainer and seal rotor and that no type of mechanical sealing action was present.

Analytical Procedures.--Garrett Turbine Engine Company used an on-line analytical train in both series of tests with the exception of test 5a. The train consisted of a FID with a glass wool liquid/vapor separation filter in the test line. The above discussed glass wool liquid/vapor separation filter was installed to prevent liquids from entering and contaminating the FID and the nondispersive infrared analyzer (NDIR) 6/ sampling lines. This allowed testing for only the gaseous contaminants in the bleed air. Ambient and bleed air were sampled and analyzed continuously for unburned hydrocarbons by use of a FID.

6/ A nondispersive infrared analyzer is used in monitoring for certain gases in the detection and control of pollution. The analyzer is an optical instrument designed to monitor the concentration of a single component (at a selected wave length) in a multicomponent stream or environment.

Carbon monoxide and CO_2 were detected by NDIR analysis, and oxides of nitrogen were determined by chemiluminescence (emission of light during a chemical reaction) analysis. Appropriate gases of known concentrations were used to calibrate and maintain calibration of the analytical instruments.

Qualitative analyses were performed by Clayton Environmental Consultants, Inc., on the Tenax-activated charcoal tubes for adsorbed materials by using GC/MS techniques and further, by employing thermal desorption followed by purging and trapping (cryogenic focusing). Known compounds ^{7/} were introduced prior to the analysis of the bleed air samples in order to control the integrity of the sample.

Samples were quantitatively analyzed by Clayton Environmental Consultants, Inc., for total hydrocarbons by GC and were reported as parts per million calculated as normal hexane (N-hexane (C_6)) as a comparative reporting standard. Oil mist samples from test 3 were quantitated as the difference of the pre-test and the post-test weights of the oil filter. Two filters were analyzed for tricresylphosphate, and samples of bleed air were analyzed for p-cresol and formaldehyde.

Equally divided parts (alliquots) of vapors and the liquid phase from the cryogenic traps were obtained by Exxon Research and Engineering Company after the traps were equilibrated to room temperature. The alliquots were analyzed in a GC using a FID and were quantitated by using organic standards that were processed similarly. Constituents were desorbed from the Tenax tube by heating the tube in the presence of a carrier gas purge. The desorbed constituents were carried directly into a GC. Desorbed materials were compared to selected organic components which had been similarly adsorbed and desorbed from Tenax traps.

In the Environmental Protection Agency's laboratory, the adsorbed constituents on the Tenax cartridges were thermally desorbed at approximately 250°C while continuously flushed with helium gas. The purged organic vapors were collected in a trap cooled with liquid nitrogen. The sample was then heated to 200°C and entered into a GC/MS which was used to separate and identify compounds that had been adsorbed on the Tenax cartridges.

TEST RESULTS AND EVALUATION

Garrett Turbine Engine Company

A tabular summation was prepared of the measured values of contaminants detected in the bleed air samples. The summation is presented in table IV. These contaminants consisted of unburned hydrocarbons, CO , CO_2 , and oxides of nitrogen. The values listed are approximate due to the very low levels present in most samples. The data plots of these measured values are located in appendix H and are designated as figures B through H, J1 and J2, K1 through K6 and L; interpretations of several of these data plots are presented in appendix G.

An evaluation of the Garrett Turbine Engine Company test data showed that there was no detectable differences in air contaminants above ambient levels during tests 2 through 3f. When the glass wool filter was removed, the unburned hydrocarbon level increased substantially without an increase in CO , CO_2 , and NO_x , indicating that the

^{7/} The terms "compound(s)" and "component(s)" are used in this report. Compounds refer to a definite chemical identity. Components are considered parts of specific mixtures.

Table IV.--Bleed air constituent elements tabular summation (ppm).

<u>Test No.</u>	<u>Test Condition</u>	<u>Figure Number</u>	<u>HC</u>	<u>CO</u>	<u>CO₂</u>	<u>NO_x</u>
1	Ambient Air	B	2*	3.8	.07	2*
2	Bleed Air No Oil	C	2.0	3*	.05	2*
3(a)	2 Lb/Hr Oil	D	2*	3.8	.06	1*
3(b)	4 Lb/Hr Oil	E	2*	3.0	0.6	1*
3(c)	6 Lb/Hr Oil	E-F	2*	3.0	0.6	1*
3(d)	8 Lb/Hr Oil	F	2.2*	3.0	0.6	1*
3(e)	10 Lb/Hr Oil	G	2.2	3.0	0.6	1*
3(f)	12 Lb/Hr Oil	H	2.4	3.5	0.6	1*
3(g)	12 Lb/Hr No Filter	J1-J2	64 ppm**	3.5	0.6	1*
4	No Seal	K1-K6	2.5	0.5	0.4	1*
5(a)	Engine Run Only	-	-	-	-	-
5(b)	Dirty Start	L	2 to 20 ppm	2.0	0.5	1*

* denotes "less than."

** indicates that the system was contaminated with oil.

hydrocarbon originated from liquid oil in the sample lines. The results indicated that essentially the same hydrocarbon levels were detected in the bleed air as in the ambient air except during oil injection into the air inlet with the in-line glass wool filter removed or during a "dirty" start. In the latter case, values of air contaminants rapidly returned to previously observed levels as the engine was run.

Clayton Environmental Consultants, Inc.

During test 3, oil was collected on the membrane filters progressively from 0.35 mg to 5.51 mg which corresponded to the graduated amounts of oil that were injected into the air inlet of the engine. The quantitative results, expressed in ppm, were obtained by GC/MS and are presented in table V below. Comparative ambient fence-line air samples were also obtained which related to test 1, tests 2 and 3, and tests 4 and 5. These values are shown in parenthesis adjacent to the applicable group of tests.

Table V.--Quantitative Results (Clayton)

Sample Test	<u>Total Hydrocarbons</u>		<u>P-Cresol</u>		<u>Formaldehyde</u>	
	ppm		ppm		ppm	
1	0.04*	(0.02)	0.03*	(0.02*)	0.01*	(0.005)
2	0.02*	(0.06)	0.02*	(0.007*)	0.009	(0.007)
3	0.03*		0.01*		0.05	
4	0.03*	(0.007)	0.02*	(0.006*)	--	--
5	2.0		0.2*		0.07*	

*denotes "less than"

All of the compounds that were qualitatively identified in the bleed air samples other than Freon were also identified in the ambient air sample.

No tricresylphosphate (para or ortho isomers) was detected in the oil mist samples (test 3 c and f) at the limits of the analytical procedure used.

The hydrocarbons obtained from bleed air samples in all tests varied from 0.02 to 2.0 ppm, expressed as n-hexane. The analyses did not indicate the presence of any cresols (possible breakdown products of tricresylphosphate) at the limits of detection for the sampling and analytical methods employed. The levels of formaldehyde (a possible oxidative product in the degradation of oil) were less than the detectable limit of 0.5 ppm of the method used.

An evaluation of the Clayton Environmental Consultant's, Inc., tests results indicated that no measurable variations occurred in the quality of the bleed air when qualitatively compared with the air entering the engine intake. Additionally, the results indicated that neither oil, nor any of its additives underwent significant degradation when passing through the engine's compressor section.

Exxon Research and Engineering Company

The extremely low concentration of the hydrocarbons that were detected prevented a positive identification of the individual hydrocarbon components. It was possible in

some cases to rule out certain materials since the materials were run as standards in the GC study.

A list of the total organic components that were collected in the cryogenic traps is presented in table VI. These components are expressed in ppb and ppm.

Table VI.--Analysis of Cryogenic Traps (Exxon)

<u>Test</u>	<u>Collected Period (Minutes)</u>	<u>Total Organic Components, ppb</u>	
		<u>ppb</u>	<u>ppm</u>
1*	60	7.2	0.007
2	90	31.6	0.032
3	15	27.6	0.028
4	90	14	0.014
5	10	2.8	0.003

* Represents analysis of front trap only.

Two components of compressor bleed air were collected in the Tenax traps: the level of these components found in the intake and the compressor bleed air were not significantly different and were identified as Freon and Methanol. Samples from tests 4 and 5 had significantly high levels of a compound with a retention time corresponding to that of the Freon which was previously used to flush out the sampling lines. No other components of significance were found.

Acrolein, benzene, toluene, and xylene were not present in any of the samples. Isopropyl alcohol and Freon TF were present and were considered artifacts of the testing and collection procedures. Analysis of material adhering to the wall of the glass wool trap was analyzed as ETO-2380 turbo oil. This indicated that unchanged oil had passed through the sampling lines.

An evaluation of the Exxon Research and Engineering Company's analytical data indicated that any ETO-2380 turbo oil decomposition products that were detected were present only in ppb concentrations. Even though the maximum compressor discharge temperature was about 726° F, the oil was exposed to this environment only for a matter of milliseconds which did not result in any appreciable degradation.

Environmental Protection Agency

The Tenax glass tubes used to adsorb materials from the air in the sampling lines were damaged during transport from EPA's facilities in North Carolina to the test site at Phoenix, Arizona. The damage was detected before testing, but the damaged Tenax glass tubes were used. The results indicated that some contamination of each sample of the Tenax adsorbent had occurred; therefore, the analytical results were questionable.

A large number of compounds were evident in the GC/MS scans. These compounds are listed in appendix G as tables XXIII through XXX. The results of test 1 showed that the major detected constituents were four branched chain alkanes, that were probably C₉ isomers. During test 2, it was noted that the Tenax cartridge appeared to be contaminated with a non-aviation lubricating oil, since more than 100 compounds were obtained. All of these components were at relatively higher concentrations than in any other samples.

The results of test 3 indicated that approximately 25 percent of the sample consisted of oxygenated compounds that were consistent with the oxidation of a hydrocarbon-type oil.

In test 4, it was noted that except for compounds with formulas C_5H_{10} and C_8H_{12} , the sample contained many compounds (toluene, n-octane, xylene) as noted in previous tests that were conducted by the Environmental Protection Agency using the Tenax adsorbers which involved diluted automobile exhaust.

The results of test 5 were similar to those obtained during test 4 but were at a lower concentration; this was probably due to the shorter compound collection time of 10 minutes versus 90 minutes.

In evaluating the Environmental Protection Agency's test data, it is apparent that the in-transit contamination of all the samples of the Tenax adsorbant caused the findings to be questionable with respect to the true representation of the actual compounds detected in the bleed air samples under study. A number of compounds were detected in the various samples; however, the analytical procedures that were employed by EPA during these tests did not provide for quantitation of the results; therefore, none of these samples were quantitated. The sampling techniques used by EPA in these tests were designed for qualitative studies of organic compounds in environmental air contamination studies. The analytical system may have been inordinately sensitive for applicability to this bleed air study; thus, the results as reported did not lend themselves to evaluation as to the quantities of compounds that were detected, or the comparison from test to test.

Toxicological Evaluation of Bleed Air Contamination Test Data

A toxicological evaluation of the test data showed that the CO concentration in the collected bleed air samples was no greater than in the ambient air. Carbon monoxide at this level of concentration was not of toxicological significance, and the fact that elevated concentrations were not found during injection of oil into the engine air intake indicated that oil passing through the compressor did not undergo degradation to an extent that significant quantities of CO were produced.

The toxicological evaluation also showed that the CO_2 concentration in the collected bleed air samples was no greater than the concentrations found in the ambient air. The quantity was not of toxicological significance. Additionally, the level of oxides of nitrogen were no greater than those in the ambient air and were of no toxicological significance.

The total level of hydrocarbons (FID-detectable oxidizable organic materials expressed as propane equivalents) that were detected in the operating engine were no greater than those in the ambient air except under two conditions:

- (1) When oil was injected directly into the air intake of the engine, almost all the hydrocarbon load in the bleed air was removed by the glass wool filter in the sampling line; this indicates that the hydrocarbons were not gaseous but were most likely in the form of an oil mist. By removing the glass wool filter from the sampling line and maintaining a 12 lb/hr oil injection rate, the FID indicated a 64 ppm normal propane (n-propane) hydrocarbon concentration in the bleed air.

- (2) When the engine was started (test 5) with oil in the air inlet, and with fuel vapors in the compressor compartment, the hydrocarbon loading at 20 ppm was rapidly dissipated. Therefore, this condition produced nothing of toxicological significance.

The cryogenic and Tenax trapping and analyses conducted by Exxon showed that the total organic components in the compressor ranged up to 31.6 ppb and that neither injection of oil into the air inlet nor removal of the seal significantly increased the total organic load of the bleed air. These findings indicate that the glass wool U-tube upstream of the cryogenic traps filtered out the mist during the oil injection test (test 3g) and that there was no significant organic load in a gaseous form. The absence of acrylonitrile, acrolein, benzene, toluene, and xylenes ^{8/} in the samples further indicates that the residual oil in the compressor did not degrade sufficiently to yield these compounds. The absence of any materials other than Freon (or methanol) and isopropanol (reasonably foreseeable potential contaminants) on the Tenax absorbers further indicated a very low (if any) gaseous load of organic materials in the bleed air of the TPE331 engine as tested.

The analyses conducted by Clayton Environmental Consultants, Inc., indicated that whether the engine was operating with or without an intact carbon seal there were no significantly measurable differences in bleed air versus ambient air. Clayton's analyses confirmed an oil mist load in the bleed air during injection of oil into the engine air inlet, but there were no indications of degradation of the oil in transit from the intake to the bleed air port. The levels of total hydrocarbons that were found in the Clayton analyses were of no apparent toxicological significance. Clayton's results further suggested that any tricresylphosphate that might have been in the bleed air was below the level of detection by the methods employed.

The analyses performed by the Environmental Protection Agency Environmental Sciences Research Laboratory did not lend themselves to meaningful interpretation because of apparent contamination of the samples and lack of parallel analysis of known compounds to permit quantitation of the various components that were revealed by the GC-MS analyses.

Based on the analyses performed, there was no significant toxicological gaseous content of the TPE331 engine bleed air under the various modes of operation prescribed by the test protocol.

Is there any unusual degree of toxicity associated with the thermal decomposition products (TDP) of ETO-2380 turbo oil? CAMI researchers exposed rats to the TDPs of ETO-2380 (new and used) turbo oils and to TDPs of other oils and found no evidence of any unusual toxicity in that the times to incapacitation and death were similar for several oils and oily materials, and the toxicological effects were consistent with the amount of CO released. The FAA Office of Aviation Medicine has published a report (FAA-AM-83-12) presenting the results of this study. The report is titled "Inhalation Toxicology: III. Evaluation of Thermal Degradation Products from Aircraft dated April 1983, and Automobile Engine Oils, Aircraft Hydraulic Fluid, and Mineral Oil.

Was there any indication of decomposition of the oil during the tests? During the engine tests a number of participants sniffed the bleed air lines that came to the sampling trains and particularly while oil was being injected into the air intake. No person reported irritation of the mucous membranes of the nose or eyes. No one described an acrid or irritating quality in the bleed air. The consensus was that the odor was that of a warm oil, not a decomposed oil.

^{8/} Benzene, toluene, and xylene are not decomposition products of synthetic turbine oils.

These observations have significance because had the oil undergone decomposition, irritating substances, such as acrolein, would have been produced, and probably would have been detected by those who sniffed the bleed air lines. The threshold odor concentration (TOC)--the concentration level at which observers sniffing the bleed air lines should detect the acrid or irritating odor of acrolein--is in the range of 0.05 to 0.1 ppm (Handbook of Environmental Data on Organic Chemicals, page 74, Karen Verschueren, Van Norstrand, Reinhold, New York 1977). The failure to detect acrolein strongly suggests that the oil did not undergo decomposition. If, on the other hand, it did decompose, major components (assuming acrolein would have been produced) were below the TOC. This observation was consistent with the absence of significant levels of the decomposition compounds by chemical analyses.

Observations during the injection of oil into the air intake of the engine suggested that it is still a mist when it enters the bleed air. This was confirmed by the finding that oil mist was filtered out by the glass wool liquid/vapor separator filter installed in Garrett's on-line analytical train; by Clayton's findings of increased amounts of oil collected on the millipore filters while increased amounts of oil were injected into the air inlet of the engine; and by observers detecting an oily smell while sniffing the bleed air lines.

Because of these findings the question was posed: Does an oil mist of ETO-2380 turbo oil have special toxic properties? To address this issue, CAMI researchers exposed rats and chickens to an oil mist of new and used ETO-2380 turbo oil for 7 hours and were unable to demonstrate any immediate or delayed behavioral change or gross pathological alteration over a period of 40 days of observation of rats and 35 days of observation of the chickens. Further, administration of ETO-2380 turbo oil into the peritoneal cavities of rats did not elicit detectable toxicological reactions.

From a toxicological point of view, the analyses and testing indicated that:

1. There is not a sufficient load of gases in the bleed air of the TPE331 engine, even with oil ingestion, to constitute a toxic threat.
2. Any oil that may get into the bleed air does not undergo a significant degradation.
3. There are no detectable quantities of supertoxic materials produced by decomposition of ETO-2380 turbo oil under the test conditions of engine ingestion and CAMI laboratory tests.
4. Oil mist of ETO-2380 turbo oil in air, as generated in the laboratory, has no unusual toxic effects on rats or chickens.

There are instances in which chronic or repeated exposure may sensitize a person to certain chemicals so that concentrations in the ppb range may later elicit an acute hypersensitivity type reaction. Barring such an extreme chemical sensitivity, there is no evidence of a toxic incapacitation factor in the bleed air of the Garrett TPE331 turboprop engine as tested.

Condensed Reviews of Findings

With respect to the bleed air contamination test results, it is apparent that the data requiring the most pertinent and critical comparison are those generated by Exxon and

Clayton, because these data were collected and analyzed by very similar methods, i.e., adsorption, dissolution, or cryogenic collection methods, followed by chromatographic and mass spectrometric analyses.

The Clayton GC/MS data as previously presented in table V indicated that test 5 (which simulates an engine start-up with oil pooling on the air intake due to a defective carbon seal) showed the maximum level of contamination of the bleed air as compared to Test 2 (normal engine operation). Total hydrocarbon content increased 1.98 ppm, p-cresol content increased approximately 0.18 ppm, and formaldehyde content increased about 0.06 ppm. The hydrocarbons and other compounds which were identified in the bleed air samples were also present in the control (blank) samples.

The Exxon data previously presented in table VI showed the organic contaminant levels from the cryogenic sample collection ranged from 0.007 to 0.032 ppm depending on the engine test performed. The GC data for samples collected in Tenax traps also showed contamination by Freon which was used to rinse sample lines and isopropanol which was used in the cryogenic traps.

A comparison of the results of test 4 to test 2 essentially indicated that contaminant levels were not increased by the absence of the front main shaft compressor carbon seal element. These extremely low levels of contaminants did not permit proper identification. Specifically, these data show a total hydrocarbon increase of 0.28 ppm and no change in p-cresol -- changes in formaldehyde concentrations were not determined.

The Garrett mobile gas analysis unit showed essentially no rise in hydrocarbon, CO, or oxides of nitrogen when comparing test 4 (no seal) to test 2 (bleed air - normal engine). Any of the measured differences appeared to be within the range of test accuracy.

FINDINGS

1. During operation of the TPE331 turboprop engine there are a number of sealing mechanisms that prevent oil from leaking from the gearbox into the engine airflow path. These tests demonstrated that the physical condition of the front main shaft carbon compressor seal element was not significant to the question of bleed air contamination.
2. The tests and analyses show that synthetic turbine oil is not likely to experience breakdown when subjected to the conditions of the compressor section in a small, low pressure ratio centrifugal compressor type engine, such as the TPE331 engine.
3. Previous tests on a large, high pressure ratio axial flow engine show that even when a large volume of oil was injected into the compressor the only compound released was a concentration of carbon monoxide that was less than the maximum permissible level of carbon monoxide prescribed in Federal Aviation Administration. (14 CFR 25.831(b2)).
4. The bleed air from the Garrett Turbine Company TPE331-10-501M turboprop engine exhibited no significant variation in relation to the quality of the air entering the engine's intake. The difference in bleed air quality as affected by the absence of a carbon seal was minimal and probably was within the range of test sensitivity.

5. Injection of heated Exxon 2380 turbo oil (ETO-2380) into the air intake of the TPE 331 engine, indicated that neither the oil nor any of its additives undergo significant degradation when subjected to mechanical action and thermal excursions while passing through the engine's compression sections.
6. Even though the TPE 331 engine compressor discharge temperature was about 720° F, the ingested oil was exposed to this environment only for a matter of milliseconds which did not result in any appreciable degradation.
7. The maximum bleed air contamination of about 2 ppm total hydrocarbons occurred during "dirty" start-up of the TPE 331 engine.
8. The compounds identified in the test samples were essentially those which appeared in the control sample. These compounds would be expected to exist in the ambient conditions of a test area located close to a commercial airport runway and to other engine test stand operations.
9. The analytical data indicated that any ETO-2380 turbo oil decomposition products were present only in parts per billion concentrations.

CONCLUSIONS

1. The results of this test program indicate that the quality of the TPE 331 engine compressor bleed air was essentially unchanged from the quality of the air that entered the engine intake. This was true for all engine operating conditions, including the absence of the forward mainshaft compressor carbon seal element. Deliberate injection of engine oil into the engine's air intake resulted in simple atomization of the oil, and no significant chemical degradation of the oil was evident at the compressor bleed air port.
2. The detected contaminants were engine oil components that were indistinguishable from the oil that was injected into the inlet. Of those contaminants that were identified through various analytical methods, the contaminants which had formed were present only in fractional part per million quantities.
3. Based on an evaluation of the Ad Hoc Committee's investigative findings and the results of these tests, the conditions within the Garrett TPE331 engine will not produce any change in the oil as it passes through the engine compressor and no toxic or anesthetic compounds are generated from the oil. Additionally, the tests demonstrated that during engine operation there are a number of sealing mechanisms that prevent oil from leaking from the reduction gearbox into the engine airflow. Finally, the physical condition of the front main-shaft compressor carbon seal element in terms of compressor bleed air contamination is not significant to the question of cabin environmental degradation.
4. No evidence was developed to support the hypothesis of pilot incapacitation due to bleed air contamination in the TPE 331 engine from ingestion of synthetic turbine oil. Pilot incapacitation due to cabin air contamination from oil ingestion in the TPE 331 engines was not a factor in the nine previously listed aviation accidents and one incident.

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January 20, 1984

APPENDICES

APPENDIX A

DETAILED DESCRIPTION OF MECHANISM OF OIL CONTAINMENT

Seal Design Philosophy

Machinery with rotating shafts require bearings and bearings require lubrication. In order to conserve the lubricant, some form of sealing must be incorporated on the rotating shafts of all machinery. Sometimes, as in turbine engines whose bleed air is utilized to provide environmental control for an aircraft's occupants, the sealing also must assure that the lubricant cannot enter the engine bleed air path.

The selection of a seal design is dependent on the mediums being sealed. Seals with solid (grease) or liquid (oil) lubricants on one side and air on the other require a mechanical seal with a stationary surface and a rotating surface in physical contact. A mechanical seal is mandatory if the oil is under pressure or if the oil level is higher than the seal when the machine is shut down.

When the bearings are lubricated by an oil spray, the sealing task is simpler since only an air/oil mist must be prevented from flowing out of the gearbox. Often a pneumatic type seal is used for this kind of sealing. In a pneumatic seal, air is caused to flow from the outside into the chamber containing the air/oil mist. Since air is flowing into the chamber through all the available paths, the air/oil mist cannot escape. A pneumatic seal does not require any contact and, thus, does not wear. A mechanical seal is also effective for sealing a chamber containing an air/oil mist.

In the Garrett TPE331 turboprop engine, both mechanical and pneumatic sealing are provided.

TPE331 Seal Design

Two seals are used in the TPE331 engine for engine oil containment and to prevent the oil from leaking from the reduction gearbox into the engine inlet and potentially contaminating cabin bleed air. The first seal is a carbon element mechanical seal. The seal's nomenclature is front main shaft compressor seal assembly; the seal and its corresponding rotor are shown in figure 9. Figures 1 and 3(a) depict how the seal is mounted in the engine. This seal consists of a stationary carbon element which is supported by a seal retainer. The carbon element is spring loaded against a steel rotor that is attached to the engine shaft. The sealing action is provided by the rubbing contact between the face of the carbon element pressing against the rotor face. Figure 10 shows the various parts of the front main shaft compressor carbon seal.

During normal engine operation, the oil is scavenged out of the engine reduction gearbox so a mechanical seal is not necessarily required during operation. However, when the engine is shutdown, oil drains from the engine walls which increases the oil level above the shaft installation level of the compressor front main shaft; without a mechanical seal, the oil would leak from the reduction gearbox, out the engine inlet and onto the ground. Therefore, a mechanical (carbon-element) seal is necessary to prevent oil leakage when the engine is not operating and is optional during engine operation.

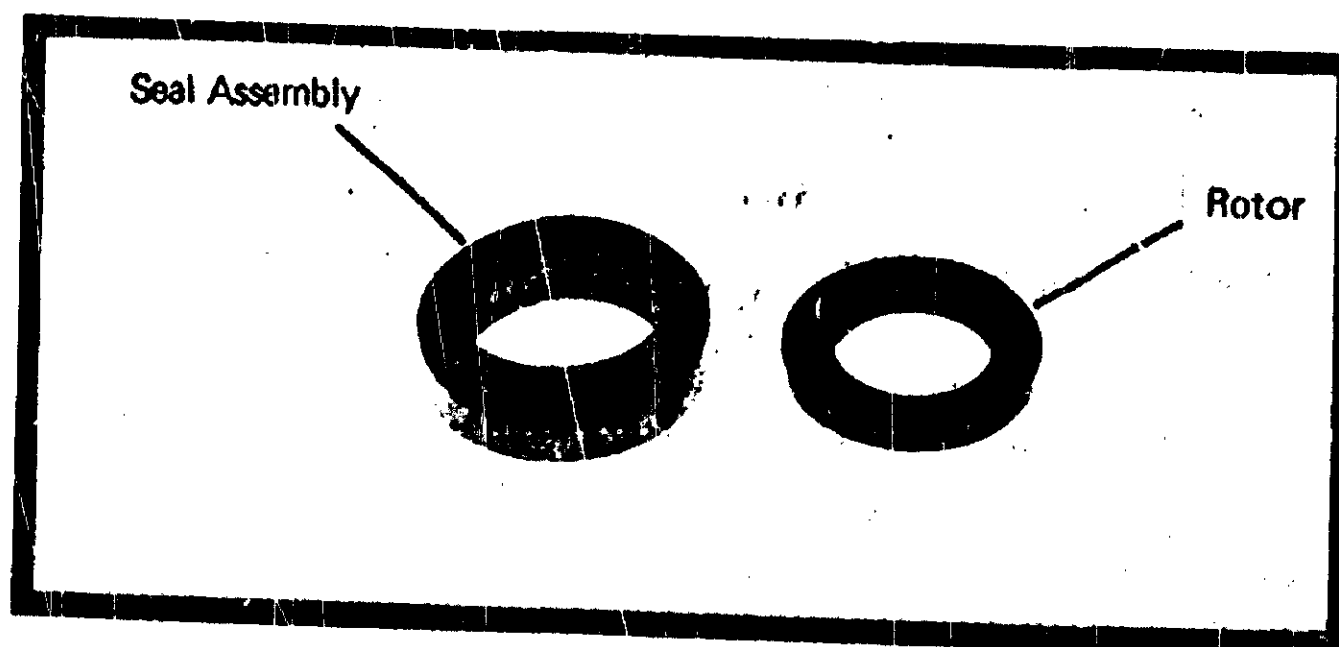


Figure 9.--Front main shaft compressor seat and rotor.

As shown in figure 3(a), the front portion of the engine shaft is supported and aligned by the compressor bearing which is positioned by a carrier that is part of the reduction gearbox housing. The seal rotor is pressed against a shoulder on the shaft by the inner race of the bearing. The seal retainer is pressed into the carrier of the reduction gearbox housing. The seal retainer carries the carbon seal element which is pressed against the rotor by a wave washer spring. An O-ring provides sealing between the carbon element and the retainer. In the drawing, the inlet air is on the left side of the seal and the reduction gearbox air/oil mist is on the right.

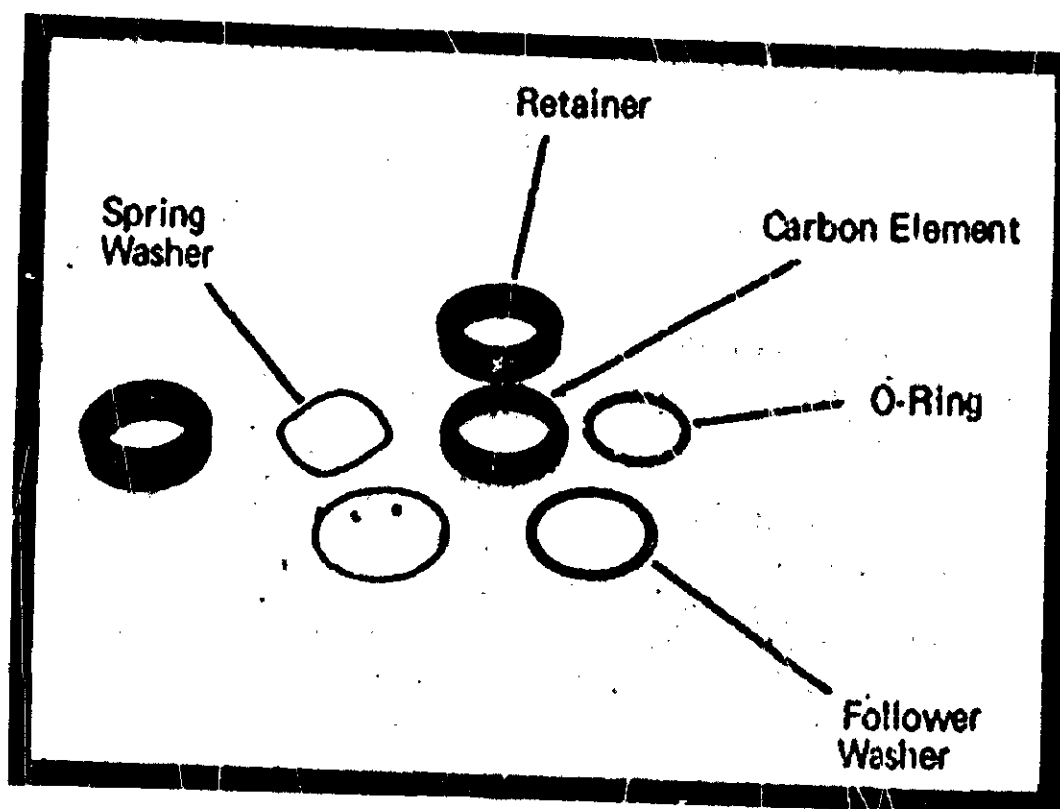


Figure 10.--Disassembled front main shaft compressor seal description.

A second independent seal, which is a pressurized rotating labyrinth seal, is provided on the shaft of the TPE331 engine between the reduction gearbox and the compressor. This pneumatic-type seal is pressurized to approximately 26 psi at the inner knife edges. When the engine is operating, the pneumatic seal is independently capable of preventing the air/oil mist from passing out of the reduction gearbox because of a flow of pressurizing air from the seal into the reduction gearbox. However, the labyrinth seal has no sealing capability to prevent engine oil from exiting the reduction gearbox when the engine is not operating and, therefore, requires a mechanical seal to prevent oil leakage when the engine is shut down. The symptom of a failed mechanical seal is oil leaking past the labyrinth seal and running out the engine inlet onto the ground when the engine is not operating. The installation of the above seal is shown in figure 11. Figure 12 shows the labyrinth seal's relative location.

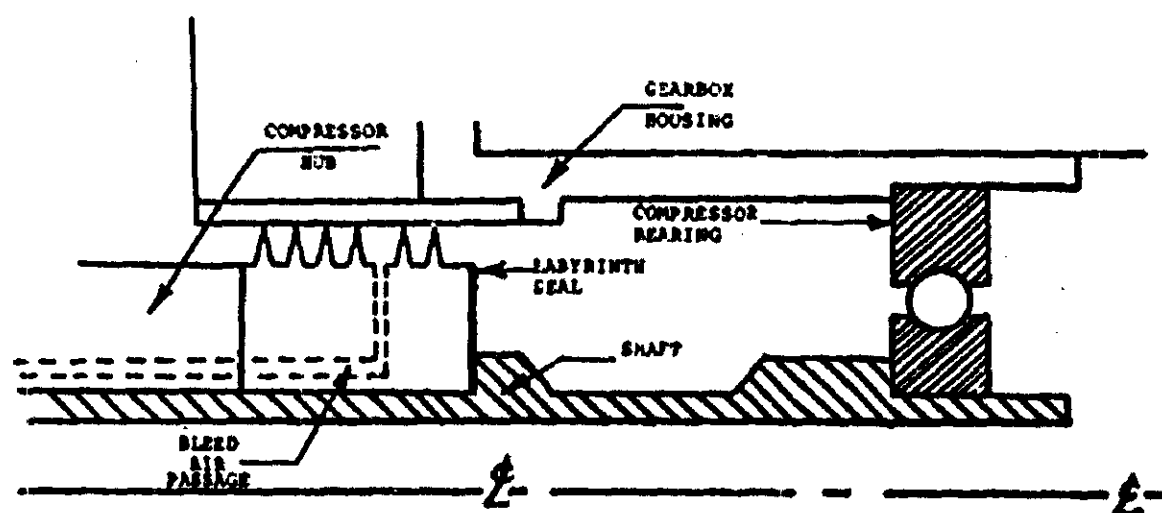


Figure 11.--Pressurized rotating labyrinth seal mounted on the engine shaft with the compressor bearing.

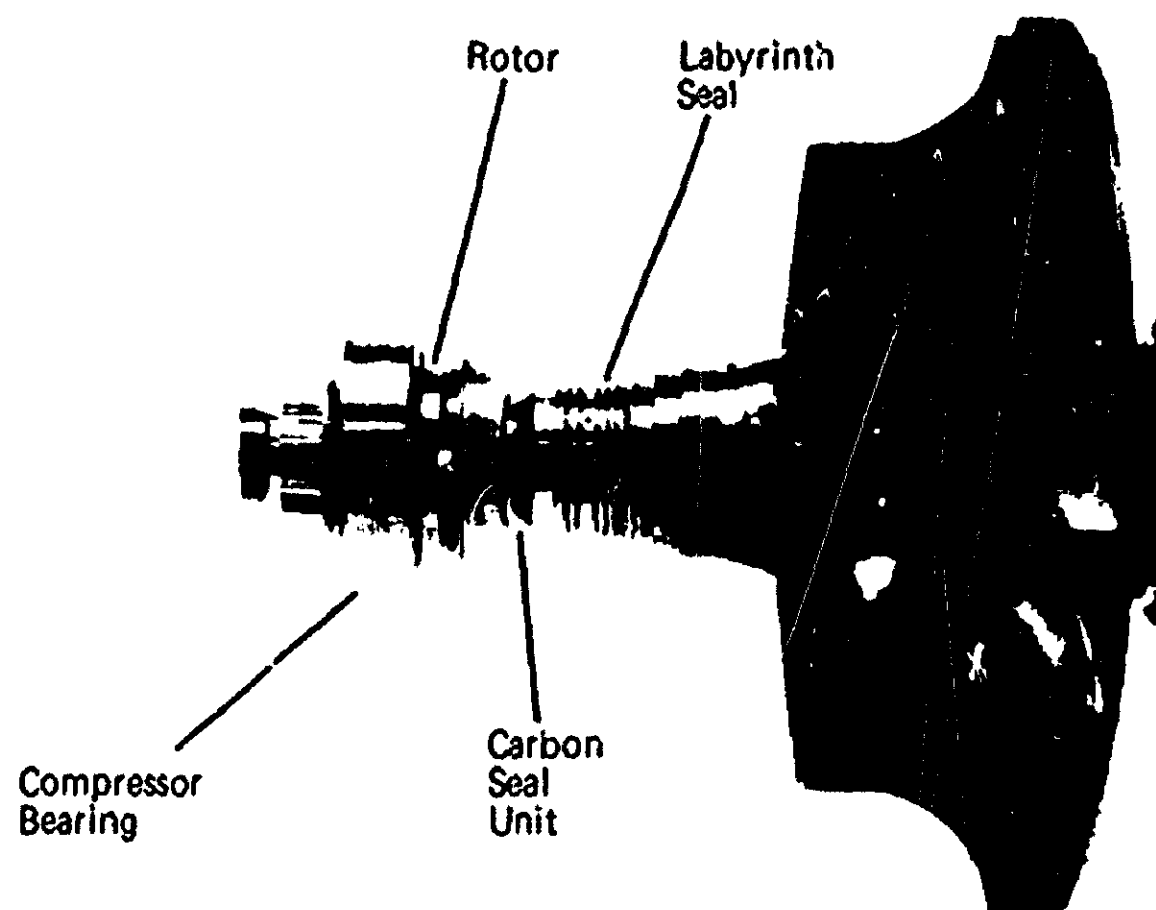


Figure 12.--Installation of the pressurized rotating labyrinth seal and front main shaft compressor seal.

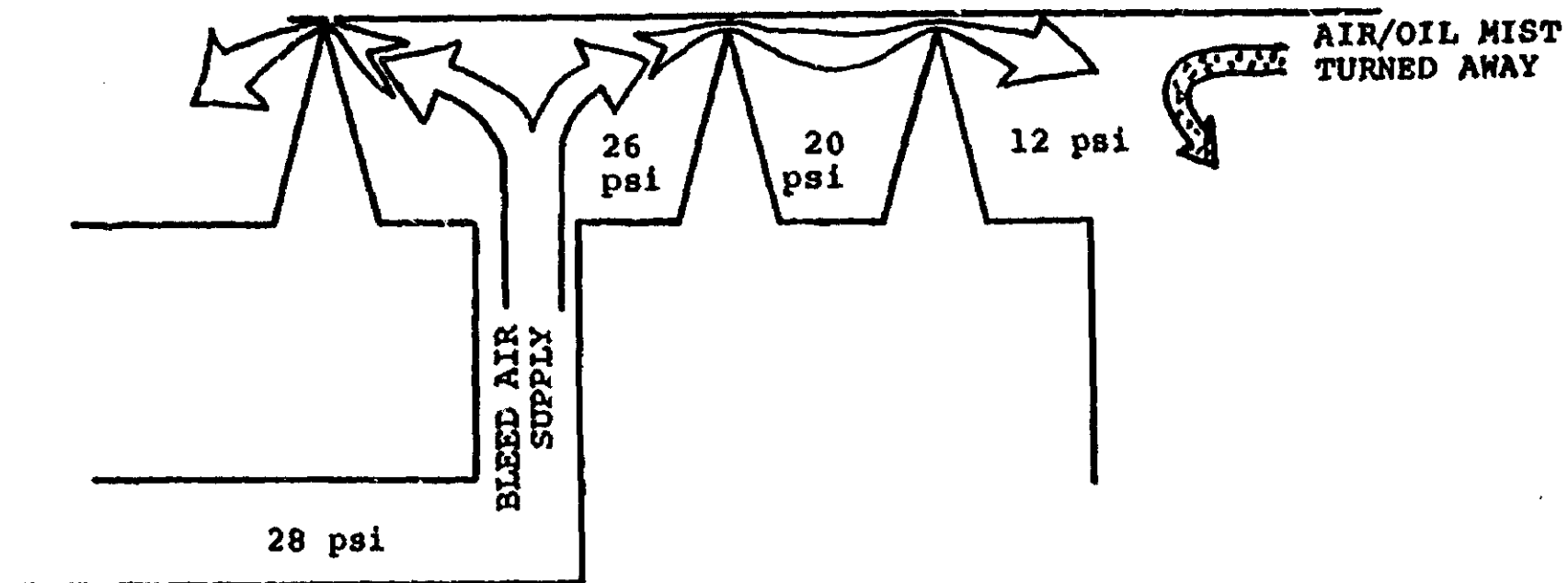


Figure 13.--Labyrinth seal pressure balances

The pressure balances that are maintained may be clearly understood by considering that the chamber containing the air/oil mist operates at approximately 12 psia (-2 psig) while the center of the labyrinth is pressurized to 26 psia (+12 psig). Since air will always flow from a higher pressure area to a lower pressure area, air will flow outward from the center of the labyrinth. Each of the knife-edges operates with only slight clearance from the outer wall, resulting in a high local velocity as the air crosses into the next chamber. With a high local velocity, the air/oil mist cannot flow past the knife edges and enter the compressor air flow. Figure 13 depicts the methods by which pressure balances are maintained by the seal.

Pneumatic Sealing Actions

In addition to the labyrinth seal, there are two other pneumatic sealing actions incorporated in the oil containment design of the TPE331 engine.

First is maintenance of a negative pressure of approximately 2 psi inside the reduction gearbox with respect to atmospheric conditions. This is accomplished by pumping the oil out of the reduction gearbox at a rate twice that at which it is being pumped in. (Nine gallons per minute in, versus 18 gallons per minute out.) The extra nine gallons pumped per minute is air and this creates a negative pressure (slight vacuum) in the reduction gearbox. If the mechanical seal should fail, the overscavenging will draw air into the reduction gearbox and will prevent the air/oil mist from flowing out. Thus, if the reduction gearbox remains intact and the reduction gearbox's housing is not breached, any resultant leakage would be of air into the reduction gearbox rather than oil out of the reduction gearbox.

Secondly, the negative pressure effect is supplemented by raising the pressure in the chamber on the air side of the mechanical seal to a positive six psi above ambient. Since the reduction gearbox pressure is continuously being reduced by the action of the scavenge pumps, a positive pressure differential will always exist, assuring that the flow will always be of air into the reduction gearbox and that the air/oil mist cannot escape.

Potential Failure Modes

Since the labyrinth seal does not contact any surface during operation, it does not wear and has no failure modes. The only condition that could cause oil to leak past the labyrinth is a bearing failure that would cause the shaft to shift out of its normal alignment causing the labyrinth to contact the adjacent wall. Such a bearing failure would result in engine shutdown either with or without pilot action. Another condition that might allow oil to enter the engine airflow would be a structural failure or penetration of the reduction gearbox housing. A propeller failure might also result in oil ingestion into the engine.

APPENDIX B

DETAILED DISCUSSION OF LUBRICATING OIL USED IN THE TEST PROTOCOL

Exxon Turbo Oil 2380 (ETO-2380) is an ester based synthetic turbo oil. The esters are synthesized from a family of alcohols known as polyols and low molecular weight fatty acids. In addition to the esters, a finished lubricant will contain antioxidants, as well as additives to prevent corrosion and tricresylphosphate which serves as an antiwear agent. ETO-2380 has a high thermal and oxidative stability and is an effective lubricant over a wide temperature range. This product has been subjected to extensive animal testing and the order of toxicity has been found to be very low.

During decomposition, synthetic lubricating oils, such as ETO-2380, may experience breakage of chemical bonds yielding compounds such as aldehydes, ketones, hydrocarbons and other molecular fragments. Depending on the temperatures involved, some oxidation of the oil molecules and their fragments might be expected to occur, releasing carbon monoxide and possibly aldehydes. Basically, up until the point when pyrolysis (decomposition due to heat stress) begins, which requires oil molecule contact with fairly high temperatures, no molecular decomposition should occur. This generally restricts the possible decomposition products to fragments and simple oxidation products of already existing molecules.

Each batch of ETO-2380 conforms to the test requirements of MIL-L-23699C and is released only after approval is given by an authorized U.S. Government Quality Assurance Representative agent. These data are on file at the Specialties Dept., Exxon Co., U.S.A. The inspection data for Batch 256 of ETO-2380 are given in figure 14; this is the batch of oil used in the Phoenix tests at Garrett Co., on July 21 and 22, 1981.

REFINING DEPARTMENT
BAYWAY REFINERY

Formula No. 2380
Product: Lubricating Oil, Aircraft
Turbine Engine, Synthetic
Base Turbo Oil 2380

Specification: MIL-L-23699C

Date 6-12-81
Order No. _____
Destination _____
Customer Order No. _____

Test Completed 6-12-81
Batch No. 256
Qualification No. 0-1-A
Tank No. 990K
Quantity Represented _____

This is to certify that a sample of the product from the above batch was tested and meets the requirements of the above specification. The composition of this batch, within manufacturing tolerance, is identical to the product qualified as a result of the qualification test. The original of this certificate is on file at the Specialties Department, P.O. Box 94, Linden, New Jersey 07036.

Para.	Test	Specification	Results
3.2	Materials - Product contains no metalorganic compounds of titanium and not more than one percent ortho isomer in the tricresyl phosphate additive.		
3.3	Viscosity, cs @ 210°F	5.0 min. 5.5 max.	<u>5.03</u>
	" " @ 100°F	25.0 min.	<u>26.12</u>
	Flash Point (COC), °F	475 min.	<u>490</u>
	Pour Point, °F	-65 max.	<u>-70</u>
	Total Acid Number	0.50 max.	<u>0.35</u>
	Viscosity, cs @ -40°F	13,000 max.	<u>7873</u>
	Evaporation @ 400°F (6-1/2 hrs.)	10 max.	<u>4.74</u>
	Foaming - Sequence		
	1 @ 75°F, ml. max.	25/none Complete Collapse	<u>35/ 3 sec.</u>
	2 @ 200°F, ml. max.	25/none 1 min. max.	<u>15/ 7 sec.</u>
	3 @ 75°F, ml. max.	25/none	<u>25/ 30 sec.</u>
	Rubber Swell		
	H-Rubber @ 158°F (72 hrs.)	5 min. 25 max. %	<u>14.76</u>
	F-Rubber @ 400°F (72 hrs.)	5 min. 25 max. %	<u>10.59</u>
★	Thermal Stability and Corrosivity at 525°F		
	Viscosity Change @ 100°F	5% max.	<u>0.0</u>
	Total Acid Number Change	6.0 max.	<u>4.35</u>
	Wt. Change Metal Specimen	4.0 mg/cm ² max.	<u>0.8</u>
	Sediment		
	Sediment (1.2 micron filter)	10 mg/liter max.	<u>2.3</u>
	Ash Filtered Material	1 mg/liter max.	<u>0.6</u>

A DIVISION OF EXXON CORPORATION

Figure 14.--Lubricating oil batch data and typical inspections (page 1).

APPENDIX B

-40-

Test	Specification	Result
Corrosion & Oxidation Stability 72 hrs. @ 400°F		
Weight Change - mg/cm ²	Fe ± 0.2 Ag ± 0.2 Al ± 0.2 Hg ± 0.2 Cu ± 0.4 -5 to +25%	<u>-0.15</u> <u>-0.29</u> <u>-0.22</u> <u>-0.07</u> <u>-0.15</u>
Viscosity Change @ 100°F	(Det. <u>31.77</u> cs)	<u>21.63</u>
Total Acid Number Change	3.0 max. (Det. <u>2.33</u>)	<u>1.98</u>
Sludge Content	50 mg/100 ml. max.	<u>0.2</u>
Trace Metal Content		
<u>Metal</u>	<u>Limit, ppm max.</u>	
Aluminum (Al)	2	<u>0.2</u>
Iron (Fe)	2	<u>0.2</u>
Chromium (Cr)	2	<u>0.0</u>
Silver (Ag)	1	<u>0.0</u>
Copper (Cu)	1	<u>0.0</u>
Tin (Sn)	11	<u>4.8</u>
Magnesium (Mg)	2	<u>0.0</u>
Nickel (Ni)	2	<u>0.0</u>
Titanium (Ti)	2	<u>0.0</u>
Silicon (Si)	Report	<u>1.3</u>
Lead (Pb)	Report	<u>0.0</u>
Molybdenum (Mo)	Report	<u>0.7</u>
Ryder Gear		
<u>No. of Determinations</u>	<u>Average Relative Rating</u> <u>(% of Ref. Oil)</u>	
2	112	<u>120.5</u>
4	106	<u>—</u>
6	102	<u>—</u>
Reference Oil Average Rating	Report	<u>2940</u>
Shear Stability:		
Viscosity Loss @ 100°F	4% max.	<u>0.7</u>
Acid Assay		
<u>Acid</u>	<u>No. of Carbon Atoms</u>	<u>Mole. Percent Limits</u>
Valeric	5	16-26
Heptylic	7	19-29
Caprylic	8	28-38
Capric	10	13-23
		<u>19</u> <u>26</u> <u>33</u> <u>30</u>

*Tests Performed By: Alcor, Inc., San Antonio, Texas

**Tests Performed By: DOD EOAPA TSC Naval Airstation, Pensacola, Florida

H. J. Kergin
 Authorized Government QAR
 6/15/81



Joseph B. Taverne
 Laboratory

DATE: 6-12-81

Figure 14.--Lubricating oil batch data and typical inspections (page 2).

Test		
Corrosion & Oxidation Stability		
72 hrs. @ 400°F		
Weight Change - mg/cm ²	Fe ± 0.2	-0.015
	Ag ± 0.2	-0.029
	Al ± 0.2	-0.022
	Mg ± 0.2	-0.007
	Cu ± 0.4	-0.015
Viscosity Change @ 100°F	-5 to +25%	
	(Det. 31.22 cs)	21.63
Total Acid Number Change	3.0 Max.	
	(Det. 2.33)	1.98
Sludge Content	50 mg/100 ml. max.	0.2
Corrosion & Oxidation Stability		
48 hrs. @ 425°F		
Pratt & Whitney Modification		
Weight Change - mg/cm ²	Fe ± 0.3	0.0
	Ag ± 0.3	0.0
	Cu ± 0.3	-0.161
	Al ± 0.3	-0.015
	Ti ± 0.3	0.0
	Hg ± 0.3	-0.023
Viscosity Change @ 100°F	+50 Max.	
	(Det. 33.41 cs)	27.91
Total Acid Number Change	Report	
	(Det. 4.42)	4.07
Trace Metal Content		
Metal	Limit, ppm max.	
Aluminum (Al)	2	0.2
Iron (Fe)	2	0.2
Chromium (Cr)	2	0.0
Silver (Ag)	1	0.0
Copper (Cu)	1	0.0
Tin (Sn)	11	6.8
Magnesium (Mg)	2	0.0
Nickel (Ni)	2	0.0
Titanium (Ti)	2	0.0
Silicon (Si)	Report	1.3
Lead (Pb)	Report	0.0
Molybdenum (Mo)	Report	0.7
Ryder Gear		
No. of Determinations	Average Relative Rating (% of Ref. Oil)	
2	112	120.5
4	106	
6	102	
Reference Oil Average Rating	Report	2240

Figure 14.--Lubricating oil batch data and typical inspections (page 3).

APPENDIX B

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<u>Para.</u>	<u>Test</u>	<u>Specification</u>	<u>Result</u>
	<u>Shear Stability:</u>		
	Viscosity Loss @ 100°F	4% max.	<u>e.7</u>
	<u>Acid Assay</u>		
	<u>No. of Carbon</u>	<u>Mole. Percent Limits</u>	
	<u>Acid</u>		
	Valeric	16-26	<u>19</u>
	Heptylic	19-29	<u>26</u>
	Caprylic	23-38	<u>33</u>
	Copric	13-23	<u>20</u>

*Tests Performed By: Alcor, Inc., San Antonio, Texas.
 **Tests Performed By: DOD EOAPA TSC Naval Airstation, Pensacola, Florida

Joseph B. Tormine
 Laboratory

Figure 14.--Lubricating oil batch data and typical inspections (page 4).

APPENDIX C

DETAILS OF ISOKINETIC SAMPLING TECHNIQUES AND EVALUATION

The committee considered utilizing isokinetic sampling techniques; however, the committee noted that sampling of the bleed air for quantitative determination of contaminant vapors or gases involves drawing a portion of the air through appropriate instrumentation or collection media at a calibrated flow rate. Vapors and gases do not undergo stratification because synthetic aviation lubricants are ash free; therefore, it would not be expected that particulate matter would form. Only in cases where stratification may be a factor, as with particulate matter of moderate diameter in a rapidly moving airstream, an isokinetic sampling technique would be necessary. While isokinetic flow rates may have been achieved during these tests, maintenance of isokinetic conditions was not considered necessary for oil decomposition product determination; therefore, no specific effort was made to maintain isokinetic conditions.

APPENDIX D

DETAILED DISCUSSION OF ENGINE INSTALLED BLEED AIR SAMPLING EQUIPMENT

An adapter, as shown in figure 5, was manufactured to provide connections from the aircraft bleed air flow control nozzle to the air sampling equipment. The adapter consisted of a 1 1/4-inch-diameter stainless steel pipe that flared up to a 1 1/2 inch-diameter chromium flash-plated black iron pipe; this iron pipe was approximately 14 inches long and ended in a standard pipe cap through which was drilled a 1-inch-diameter orifice. The orifice was installed in order to create back pressure within the 1 1/2-inch pipe sufficient to force the air mixture into the sample pickup probes, through connecting lines to the sample collectors located outside the test cell.

The adapter, by design, included features to insure mixing of the bleed air with any entrained contaminants prior to pickup by the sampling probes. These features included a mitre bend downstream from the sonic nozzle followed by a straight section of 1 1/4-inch-diameter stainless steel pipe for dissipation of turbulence. The step from the 1-1/4-inch to the 1 1/2-inch diameters was made sharp edged in an attempt to cause liquid oil on the sampling pipe's wall to be atomized. Following the step, the 1 1/2-inch-diameter pipe continued undisturbed for six diameters for further turbulence dissipation before pickup by the sample probes.

Four sample pickup probes were installed radially into the 1 1/2-inch diameter pipe beginning approximately 12 inches downstream from the leading edge of the pipe. These probes were positioned 60° circumferentially, with 1-inch axial spacing. This configuration was chosen to assure minimum interference between the probes. Each probe had six equally spaced 0.062-inch holes drilled across the 1 1/2-inch length facing upstream.

The above described entire sampling system configuration was based on Volume 43, No. 43 of the Federal Register.

APPENDIX E

DETAILED DISCUSSION OF ENGINE BLEED AIR TEST EQUIPMENT, SAMPLING SYSTEMS, AND PROCEDURES

Garrett Turbine Engine Company

The emissions analysis equipment (mobile gas analysis unit) and all required calibration gases are installed in a mobile emissions analyzer van. All instruments, calibration gases, and span gases are kept at a constant temperature to avoid instrument drift. The equipment is capable of monitoring carbon monoxide (CO), carbon dioxide (CO₂), and any oxides of nitrogen (NO_x), including nitric oxide (NO) and any unburned hydrocarbons in the air. This on-line instrumentation continuously measures the resultant sample flows. The data results are graphed on a four-channel recorder for permanent records.

The primary emissions measuring equipment used by Garrett, consisting of the Flame Ionization Detector (FID) and Nondispersive Infrared Analyzer (NDIR) measurement equipment, was operated continuously throughout the test series. These measurements were recorded continuously on a strip chart that gave a complete history of the tests.

The equipment is capable of determining whether any change is taking place in the oil as it passes through the engine. If oxidation is taking place, then a rise in the carbon monoxide and/or carbon dioxide level will occur. Any other change to the oil that occurs will release volatile compounds that will be detected by the FID unit as unburned hydrocarbons.

Ambient and bleed air sampling were conducted for total unburned hydrocarbons by flame ionization detection; carbon monoxide and carbon dioxide were detected by nondispersive infrared detection, and oxides of nitrogen by chemiluminescence analysis.

Unburned hydrocarbons were measured with a Beckman Model 402 flame ionization detector (FID). This system has the capability of detecting any ionizable hydrocarbons as propane, with concentrations as low as 0.1 - 0.2 parts per million (ppm); however, the system does not have the specific capability to identify what chemical compounds (by-products) are present in the hydrocarbons. The FID is calibrated at 0-10 ppm and 0-100 ppm. The unit accuracy is 1 percent of full scale for both of the above calibrations at this setting.

Carbon monoxide and CO₂ were measured with Beckman's Model 315B NDIRs. The CO₂ unit is calibrated at 0 to 2 percent concentration and has ± 1 percent of full scale accuracy. The CO unit is calibrated at 0-100 ppm and has ± 2 percent of full scale accuracy.

Oxides of nitrogen measurements were made with a TECO Model 10A chemiluminescent analyzer that utilizes a thermoconverter for the NO_x mode. This equipment has a range of 0-100 ppm with a ± 2 percent of full scale accuracy.

Evaluation of the PID and NDIR data results can also determine if any change will occur in the oil when it passes through the engine. This was accomplished during the oil injection tests. On the first run, a glass wool liquid/vapor separation filter was used to remove any liquid contaminants from the bleed air. An oil injection test was then performed, while an analysis was conducted for gaseous contaminants in the bleed air. During the run, propane (C_3H_8) was momentarily injected into the engine inlet to momentarily contaminate the bleed air. This confirms that if any gaseous contaminants are present they will be detected by the system with the separation filter installed.

A 7-foot insulated stainless-steel line was connected to the sample probe at the engine bleed air pickup. This line was in turn connected to a 20-foot heated line whose temperature was controlled at approximately 250° F by a variac type control. A second 20-foot heated line, whose temperature was measured and controlled from the mobile analyzer van, directed the samples into the testing equipment. Except for test 3(g), the oil injection test, the temperature was held at 250° F \pm 5° by an automatic controller.

A typical calibration record of the Garrett sampling equipment is shown in figure 14. The following calibration values, as shown in table VII, below used.

Table VII.--Garrett bleed air sampling equipment calibration values.

	<u>Full Scale</u>	<u>Calibration Level</u>
Unburned Hydrocarbons	100 ppm	50.3 ppm
Carbon Monoxide	100 ppm	60 ppm
Carbon Dioxide	1.05%	2.0%
Oxides of Nitrogen	100 ppm	46.8 ppm

The full scale values shown above were used throughout the testing with the exception of test 4 (compressor seal carbon element removed) where the scale for unburned hydrocarbons was expanded 10X to give a full scale value of a 10 ppm.

Clayton Environmental Consultants, Inc.

Separate samples of ambient and bleed air were collected using combinations of solid and liquid sorbent media. The test sampling system is shown in figure 6 and a schematic representation of the system is presented as figure 7.

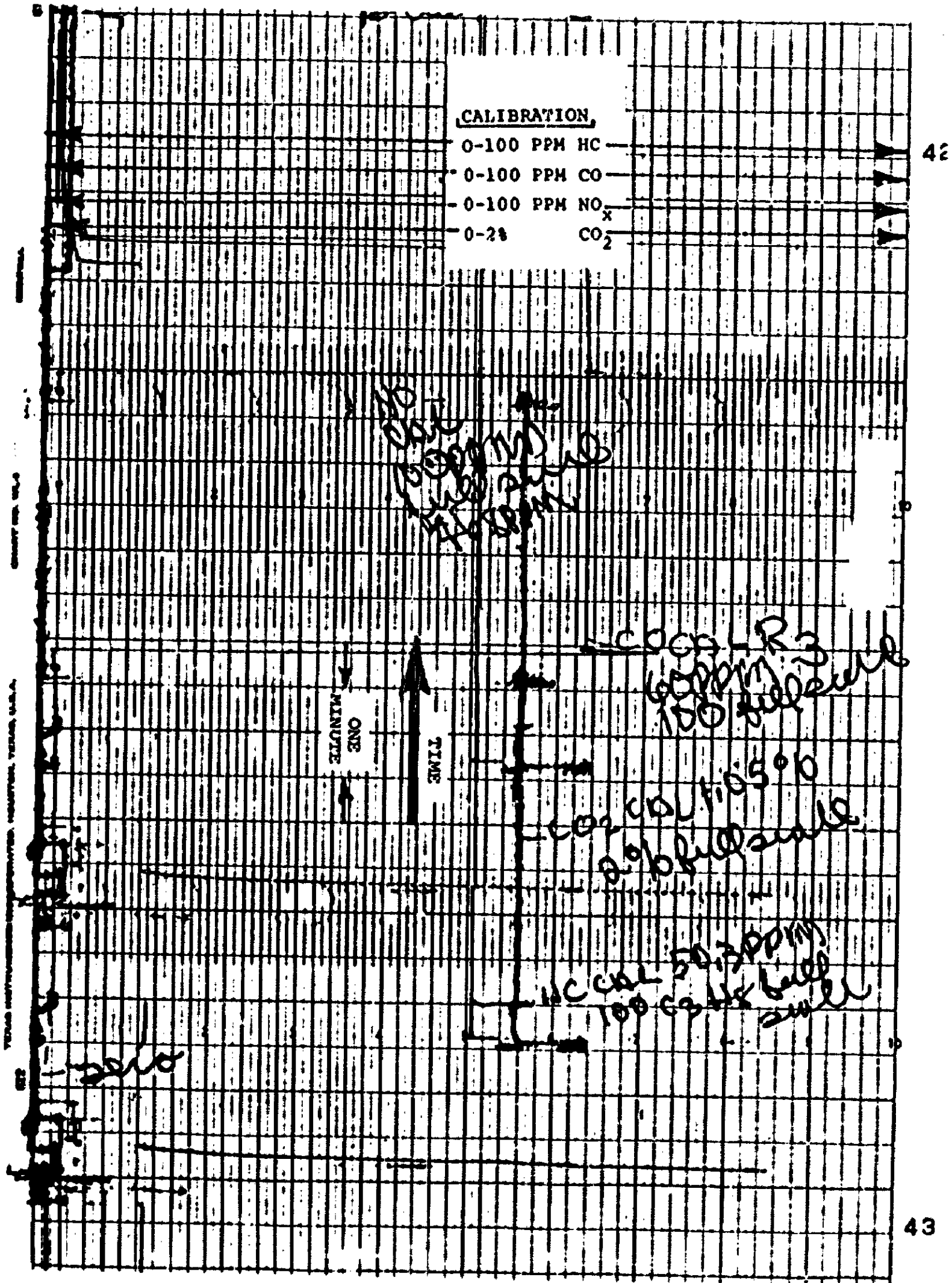


Figure 15.--Typical calibration record Garrett bleed air sampling equipment.

The testing methods used by Clayton for obtaining bleed air samples for test No. 1, engine data points 1, 2, 3, and 4, consisted of drawing engine inlet air through a stainless steel tube from behind the propeller to pickup ram air. The samples were then transmitted via stainless steel (approximately 30 feet), and teflon (approximately 0.5 foot) tubing to a glass manifold equipped with five outlet ports. Separate air samples were drawn from four of these ports while the fifth was used for manifold pressure control.

Solid sorbent media, e.g., Tenax and activated carbon (ACT) were selected to cover the range of possible organic compounds. Higher sampling flow rates were selected to achieve the low detection limits. Duplicate samples were collected to allow compound identification using gas chromatography/mass spectrometry (GC/MS); quantification of these compounds was accomplished by using GC.

Working forward from the manifold inlet, the outlet ports were each connected via teflon fittings to the following sample trains:

Port 1: Two glass tubes in series, the first packed in two sections with 150 milligrams (mg) of Tenax solid sorbent medium and the second packed in two sections with 150 milligrams of activated charcoal. These sorbent tubes are products of SKC, Inc. Air was drawn through the sorbent tubes using an E.I. Dupont de Nemours Company Model P-125 portable battery-powered personal sampling pump. The pump was calibrated during the test using a soap bubble technique with a burette attached to the pump's discharge port.

Port 2: One glass tube packed in two sections with 150 mg of Tenax solid sorbent. Air was drawn using a portable pump as in (1) above.

Port 3: One glass tube packed in two sections with 150 mg of activated charcoal. Air was drawn using a portable pump as in (1) above.

Port 4: A 6-inch section of tygon tubing connected to two midjet impingers arranged in series. Each impinger contained approximately 15 milliliters (ml) of a 1-percent sodium bisulfite absorbing solution. The impinger train in turn was connected via tygon tubing to a Mine Safety Appliance Company Model G portable battery-powered personal sampling pump. The pump was calibrated prior to the test program and was again calibrated using the soap bubble-burette technique at the conclusions of the tests.

Port 5: Fitted with tygon tubing running through a bleed valve to a U/tube water manometer. Manifold pressure was controlled using the bleed valve and monitored on the manometer. Approximate gas temperature was monitored by inserting a bimetal dial thermometer into the exhaust from the bleed valve.

Simultaneous with the inlet air sampling, ambient air was sampled at a fence that was located due east of, and about 20 feet from the test engine. The sampling media and pumps were of the same type as previously described, although no manifold was used. Ambient air sampling was conducted for correlation with the inlet air sampling tests. This ambient sampling was conducted during the entire test period. Following Test No. 1, and for the remainder of the testing period, the Tenax in series with charcoal collection method was omitted from the ambient air sampling station.

Tests 2 through 5 involved sampling of engine bleed air. A specially fabricated sample collector (described earlier in this report) was installed on the engine. From this collector, bleed air was transported to the Clayton manifold as described above, via an unheated stainless steel line.

Testing methods used during test Nos. 2, 3g, 4, and 5b, consisted of drawing samples of engine bleed air from the glass manifold. In each of the above tests, separate samples were drawn through a Tenax-activated carbon sorbent series, a Tenax sorbent tube, an activated carbon sorbent tube, and a 1 percent sodium bisulfite filled impinger train as described for test 1. Sampling flow rates were controlled using the battery-powered personnel sampling pumps. During the tests, a second bleed valve was installed upstream of the glass manifold to assist in holding manifold pressure within desired ranges. Manifold pressure was monitored by a water gauge manometer, attached to the fifth port.

Tests 3a, b, c, d, e and f, comprised a special series intended to determine the content, if any, of oil mist contamination in bleed air when oil is injected into an operating engine's air intake. The previously described bleed air collection equipment and stainless steel line were used. A bypass valve was installed at the outlet of the stainless steel sample delivery line; this valve was used to control line pressure going to a millipore membrane filter located downstream of the bypass valve. This pressure was maintained at approximately 3 inches of water, as measured by a monometer which was momentarily substituted for the filter for pressure measurement between each test. Samples were drawn from the line downstream of the bleed valve through separate 37-millimeter millipore membrane filters, one for each test identified above. The filter was composed of mixed cellulose ester medium with an average pore size of 0.8 micrometer. The filter was housed in a plastic cassette, the cassette intake was fitted to the sample line, and the exhaust was connected to an MSA Model G personal sampling pump.

Oil mist samples were collected by drawing air at measured flow rates through preweighed 37-mm mixed cellulose ester membrane filters using an MSA, Model G battery-powered portable pump. The total mass collected on each filter was determined gravimetrically in the laboratory as the difference between the tare weight of the filter and the weight of the filter after sampling and equilibration to balance room conditions. Results of the gravimetric analysis, used to calculate the total weight gain include necessary corrections for blank determinations run in parallel with the analyses.

The analyses for oil mist was conducted by emission fluorescence using Physical and Chemical Analytical Method (P & CAM) No. 159 as specified by the National Institute of Occupational Safety and Health. In this method, the calibration standards are prepared from bulk samples of the oil taken at the time of sampling. The proper excitation wavelength was determined using a 100 microgram per milliliter solution of the oil in chloroform. After extracting the filter matrix with chloroform the extract was scanned over a range, including the predetermined wavelength. The quantity of oil was then determined by comparing the peak-height of the sample with the peak-height of each standard. After any necessary corrections for comparative control (blank) samples run in parallel with the samples, the results were reported as total milligrams of the specific oil per sample.

Total hydrocarbons samples were collected by using battery-powered, portable pumps to draw air at measured flow rates through glass tubes containing activated charcoal (containing 100-milligram (front) and 50-milligram (back) sections of activated charcoal).

Immediately after sampling, the ends of the tubes were sealed with polyethylene caps for transport to the laboratory, where they were refrigerated until analysis.

In the laboratory, front and back sections of each tube were desorbed separately in appropriate volumes of carbon disulfide and aliquots of the resultant solutions injected into a Hewlett-Packard 5710A gas chromatograph. Because the samples contained a complex, nondescript mixture of hydrocarbons of varying molecular weights and structures, quantification of specific components was not possible. Quantities of total hydrocarbons present were determined by comparison of the total of the areas under the sample chromatogram peaks with areas under chromatogram peaks for n-hexane standards prepared in carbon disulfide. Analytical results, which include any necessary corrections for parallel blank determinations, were used in conjunction with sampling data (volume of air sampled) to calculate the concentrations of airborne total hydrocarbons (as hexane) represented by each sample, expressed in parts of vapor per million parts of air by volume (ppm).

Ortho and para cresol (o-cresol and p-cresol) samples were collected by using battery-powered, portable pumps to draw air at measured flow rates through glass sample collection tubes containing the appropriate solid sorbent in two sections (front and back).

The ends of tubes were sealed immediately after sampling with polyethylene caps for transport to the laboratory, where they were refrigerated until analysis.

In the laboratory, front and back sections of each tube were desorbed separately in appropriate volumes of acetone, and aliquots of the resultant solutions injected into a gas chromatograph. Quantities of each analyte present were determined by comparison of areas under the sample chromatogram peaks with areas under chromatogram peaks for standards prepared in acetone. Analytical results, which include any necessary corrections for parallel blank and recovery determinations, were used in conjunction with sampling data (volume of air sampled) to calculate the concentrations of o-cresol and p-cresol represented by each sample, expressed in parts analyte per million parts of air, by volume (ppm).

Tricresylphosphate samples were collected using MSA Model G battery-powered portable pumps to draw air at measured flow rates through preweighted 37 mm mixed cellulose ester membrane filters. The total mass collected on each filter was determined gravimetrically in the laboratory as the difference between the tare weight of the filter and the weight of the filter after sampling, and equilibration to balance room conditions. Results of gravimetric analysis used for comparison against tricresylphosphate (TCP) contents include necessary corrections for blank determinations run in parallel with the analyses.

For the analysis of separate isomers of polycresylphosphate, the filters were treated in appropriate volumes of ether, and aliquots of the resultant solutions were injected into a gas chromatograph equipped with a nitrogen/phosphorous specific detector. Quantities of each analyte present were determined by comparison of areas under the sample chromatogram peaks with areas under chromatogram peaks for standards prepared in ether.

Analytical results, which include any necessary corrections for parallel blank and recovery determinations, were used in conjunction with sampling data (volume of air sampled) to calculate the concentrations of airborne isomers of tricresylphosphate represented by each sample, expressed in parts tricresylphosphates per million parts of air (ppm) by volume.

Formaldehyde samples were collected by drawing air at measured flow rates (nominal 1 liter per minute) through all-glass midjet impingers containing 15 ml of 1 percent sodium bisulfite absorbing solution using battery-powered, portable pumps. After collection, the impinger solutions were immediately transferred into glass bottles and sealed with teflon lined caps for transport to the laboratory.

Laboratory analysis was accomplished by treating aliquots of each sample with aqueous chromotropic acid and concentrated sulfuric acid to form a colored complex whose absorbance ^{9/} was measured using a spectrophotometer. The absorbances of the samples were compared to the absorbances of aqueous standards prepared from sodium formaldehyde bisulfite and carried through the same analytical procedure. Results of the analysis in conjunction with sampling data were used to calculate the concentrations of airborne formaldehyde (expressed in ppm) and include any necessary corrections for blank and recovery determinations run in parallel with the analysis.

Exxon Research and Engineering Company Condensed Review Discussion

A photograph of the Exxon Product Research Division (PRD) bleed air sampling train is shown on figure 16. A schematic representation of the bleed air sampling train is shown as figure 8.

Two methods were used to collect bleed air samples. The first method used was a cryogenic process. The second method consisted of a Tenax adsorbent material that was used to trap contaminants from the air stream being sampled.

The first method was previously used by Exxon in their continuing Aviation Lubrication Project. The project is designed to investigate oxidative mechanisms. In this method, the air from the compressor bleed air system was passed through traps that were in the air sampling train. The air passing through these traps was chilled at -78° C (-108° F) in a dry ice/isopropyl alcohol train; the two traps were then analyzed for three of the tests and only one for the background test. The second tube of test No. 5 (Dirty Start Test) was broken during shipment. All samples which were collected in the freeze out train were carefully weighed and stored in dry ice until they were analyzed by Exxon's Analytical and Information Division. The collected samples were analyzed by means of a GC coupled with a flame ionization detector. In addition to the Cryogenic Method freeze out train, each engine test was also monitored with a Tenax Column for the second test. This is a small tube about 1/4 inch in diameter and 6 inches long which has its own pump and counter to measure the air through the solid adsorbent. These columns are subsequently desorbed by heat into a GC/MS to measure the adsorbed contaminants. The Tenax columns are much simpler to handle than the freeze out method and are used widely for pollution studies.

A schematic drawing of the sampling train is presented as figure 8 and should be referred to in the following description.

^{9/} In a physics definition - one measure of the degree to which a solution absorbs radiant energy.

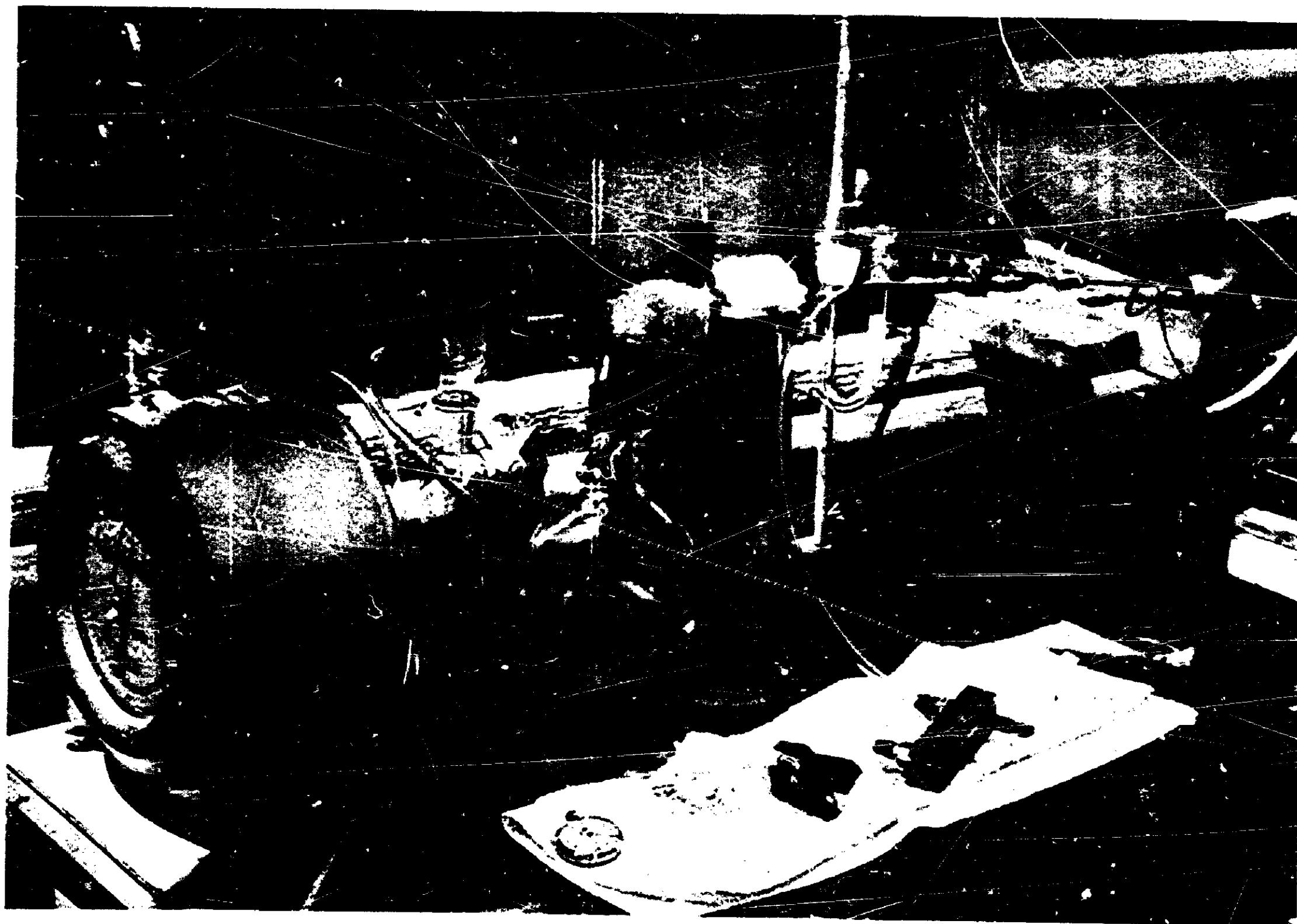


Figure 16.--Exxon bleed air test sampling system.

The 1/4-inch-diameter stainless steel sample line from the engine was connected to a Y-adapter which was fitted with two Hoke needle valves. This provided independent control of the source flow rate for the two sampling methods. One needle valve was fitted with a stainless steel ball joint adapter. This was connected to the cryogenic sampling system which consisted of a glass U-tube containing loosely packed glass wool, two glass traps of the type commonly used in vacuum systems, a glass drying tower containing Drierite, and finally a wet test meter which was used to measure the volume of air sampled in each test. The second needle valve was connected to a stainless steel tee. One leg of the tee was connected to a rotometer which measured the flow rate of air to be sampled as it passed through the tee. The remaining leg of the tee was connected to a Tenax packed column which in turn was connected to an air sampling pump which pulled the sample through the Tenax column at approximately 1/3 cubic foot per hour. This arrangement was chosen to provide a near atmospheric pressure source for the Tenax column to the sample.

The sampling train was set up as shown in figure 8. A new glass U-tube was used in each of the first three runs. The U-tube was omitted in runs 4 and 5.

The metal Dewar flasks containing the front and rear traps were charged with dry ice and isopropyl alcohol and allowed to equilibrate. The sampling was started on cue by opening the two Hoke needle valves and turning on the air sampling pump for the Tenax column. The flow through the cryogenic system was then adjusted to approximately 10 cubic feet per hour using the needle valve. The flow in the line sampled by the Tenax column was adjusted to approximately 5 cubic feet per hour by a needle valve on the rotometer.

After the sampling period the Hoke needle valves were closed and the air sampling pump was turned off. The glass U-tube, if present, was sealed, removed from the line, and stored at ambient temperature. The front and rear traps were removed from the line, capped, and stored in a chest at dry ice temperature (-78°C). The Tenax column was removed from the line, capped, and stored at ambient temperature. The sampling train was then prepared for the next run.

The above procedure was used for runs 2 through 5 where the sample line from the engine provided sample air at a pressure of approximately 3 psi. In run 1, the sample line from the engine provided intake air at approximately atmospheric pressure. Therefore, it was necessary to pull the sample through the cryogenic collection system by attaching a vacuum pump to the outlet of the wet test meter. In the Tenax sampling system, the leg of the tee normally attached to the rotometer was capped during this run.

During the test, Exxon collected 10 cryogenic samples in glass traps cooled in dry ice and isopropanol and 5 Tenax adsorption traps. These samples were returned to Linden, New Jersey, and submitted to the Analytical and Information Division (AID) for analysis using GC procedures for quantification of the organic components collected in the various traps and GC/MS methodology for qualitative identification of the measured components.

Quantitative analyses of the contents of the cryogenic and Tenax traps were performed using GC procedures. Qualitative analysis by GC/MS techniques was also attempted on selected vapor and aqueous phases from the cryogenic traps and on the contents of Tenax trap from test 3g.

Cryogenic Traps

The organic components of compressor bleed air were collected in 95 ml glass cryogenic (freeze-out) collection traps immersed in a dry ice/isopropanol bath. These collection traps were capped with glass ball joint stoppers lubricated with methylsilicone grease and maintained in dry ice until the time of analysis. At the AID laboratory, the glass stopper on the exit arm of the tube was replaced with one equipped with a septum seal for sampling. The traps were allowed to equilibrate to room temperature (approximately 25°C). Under these ambient conditions, the tubes contained both an aqueous liquid and a vapor phase above the liquid. The vapor phase above the liquid was sampled from the collection tube through the septum with a 5.0 ml (cc) hypodermic syringe. A 3.0 cc aliquot was analyzed in a Perkin-Elmer Model 900 GC using a flame ionization detector and analyzed according to the conditions described in table VIII.

Table VIII.--Operating conditions for the gas chromatographic analyses of vapor and aqueous phases in the glass dry ice/isopropanol traps collected at the Garrett Turbine Engine Company.

Instrument:	Perkin-Elmer Model 900 equipped with a flame ionization detector
Column:	10' x 1/8" O.D. stainless steel column packed with 20 percent SP-2100 methylsilicone fluid on 80-100 mesh Supelco Port
Carrier:	Helium at 30 cc/min.
Temperatures	
- Injector:	340° C
- Detector:	350° C
- Column:	40° C for 1 minutes, programmed at 8° C/min. to 250° C final temperature
Sample Sizes	
- Vapor:	3.0 cc
- Liquid:	2.0 microliters

Following the vapor phase analysis, the aqueous phase was sampled with a 10 microliter hypodermic syringe. A 2.0 microliter aliquot of the aqueous phases was analyzed directly according to conditions described in table VIII.

The concentration of the organic components found in the various phases were calculated using an external standard of benzene in the corresponding phase. Vapor phase standards consisted of 3.0 cc blends of 4.6 ppm benzene in helium. Aqueous phase standards used a blend of 4.6 microgram/ml benzene in distilled water.

Tenax Traps

Tenax traps used to adsorb organic components from the compressor bleed air in specified experiments were capped with appropriate Swagelok^R caps and were transferred to AID for analysis. At the time of analysis, the Tenax traps were placed in the carrier gas line of the chromatograph using Swagelok^R connectors, purged with helium and then heated to 250° C with a cartridge heater and a Variac^R voltage regulator. The conditions of analysis are presented in table IX.

Table IX.--Operating Conditions for the gas chromatographic analyses of the absorbed vapors of the Tenax traps collected at the garrett turbine engine company.

Instrument:	Perkin-Elmer Model Sigma 2 equipped with a flame ionization detector
Column:	10' x 1/8" I.D. stainless steel column packed with 15 percent Carbowax 20M on 60-80 mesh Chromosorb W
Carrier:	Helium at 30 cc/min.
Temperatures	
- Injector:	175° C
- Detector:	175° C
- Column:	50° C initial; programmed at 6° C per minute to 150° C
Sample Sizes	The entire Tenax tube installed in the carrier gas line and heated to 250° C to desorb the adsorbed components

Contents of these traps were quantified using an external standard method in which Tenax traps similar to those used for analysis were fortified with selected organic components and purged with 5 liters of ambient air to simulate sampling conditions.

Qualitative Analysis

The organic components of selected vapor and aqueous phases were subjected to a qualitative analysis GC-MS by both a Finnigan Model 4000 GC/MS data acquisition system and a Perkin-Elmer Model 900 GC equipped with an effluent splitter interfaced with a flame ionization detector, and a Dupont Model 21-491 mass spectrometer were employed.

Tentative qualitative identifications of components were also performed by comparison with retention times of known organic compounds. Since more than one organic component type can exhibit the same retention time under the conditions of analysis, this method cannot offer positive, definitive identification of the suspected organic components.

Environmental Protection Agency

Ambient and bleed air samples were obtained by chromatographic adsorption using Tenax cartridge in series. These samples were analyzed using the technique of thermal desorption GC/MS microprocessor system to separate and identify the vapors trapped in the Tenax cartridges.

Teflon tubing of approximately 6 feet in length and 1/8 inch outside diameter was used to connect the Tenax sampling cartridge to the stainless steel tube that was mounted behind the propeller. A Brooks "flo-rate" rotometer and a Nupro needle valve were inserted between the pump and Tenax cartridge to maintain a constant air flow. The rotometer was calibrated in the EPA laboratory prior to sampling and again after the sampling was completed. A Thomas Model 10TCA187PB diaphragm pump was used to draw the sampled air through the Tenax cartridge.

Tests 2, 3g, 4 and 5b involved sampling of the engine bleed air, which was collected off of the same fabricated sample collector, installed on the engine, as used by Clayton Environmental Consultants. The samples were collected in the same manner as for test 1. All the samples were collected using a flow rate of 1 liter/min. The test system is shown in figure 6.

The Tenax cartridges used for sampling the bleed air were prepared for EPA by the Northrop Corp. The procedure for preparing clean Tenax cartridges involved extracting the Tenax with methanol and pentane for approximately 1 day for each solvent and baking the air dried Tenax overnight in a vacuum oven. The cartridge was prepared by adding 250 mg. of Tenax to a precleaned pyrex glass tube. The Tenax was held in place in the glass tube by methanol and pentane extracted glass wool. The cleaned cartridges were stored in precleaned culture tubes wrapped with aluminum foil and placed in a tightly sealed metal container.

The air samples collected in the Tenax cartridges were analyzed using the technique of thermal desorption GC/MS. A Nutech Model 320 thermal desorption system was used to desorb trapped organic vapors from the cartridge. The sampling cartridge was heated to approximately 250° C while being flushed continuously with a 15 cc/min flow of helium. The purged organic vapors were collected in a liquid nitrogen trap which was then heated to 200° C in approximately 20 seconds to inject the sample as a discrete plug into the GC/MS system.

A Hewlett Packard Model 5992A GC/MS microprocessor system was used to separate and identify the vapors trapped in the Tenax cartridges. The gas chromatographic column used for this analysis was a Quadrex 100m x 0.05 mm containing 7.5 percent Hydrophobic silica. The column was temperature programmed from 20° C (initial hold time 15 min) to 150° C at 4° C/min. Helium was used as the carrier gas with a flow rate of 2 cc/min. The interface between the GC and MS consisted of a thick wall small bore capillary tube which allowed 0.75cc of the GC effluent to flow directly into the MS.

The MS parameters were as follows:

Scan speed 690 amu/sec. Scan range 25 to 250 amu. Ionization voltage 70eV.
Electron Multiplier 2,000 to 2,200 volts.

A cassette tape cartridge was used to operate the GC/MS system and store the collected data. The cassette tape has a limited storage capacity and therefore only one hundred mass spectra (one mass spectrum per one gas chromatographic peak) could be obtained for each sample. If there are more than 100 compounds (gas chromatographic peaks) detected in a sample, no mass spectrum will be obtained after the 100th peak.

The mass spectral interpretations were performed mainly by comparing the spectra obtained in each sample with the spectra contained in the Registry of Mass Spectral Data (B. Stenhagen, S. Abrahamsson, and F.W. McLafferty, editors).

APPENDIX F

DETAILED DISCUSSION OF TEST RESULTS

Garrett Turbine Engine Company

During test 1, the ambient conditions were determined by sampling the propeller airflow beside the engine. This gave an indication of the contaminant level of the air entering the engine. This allowed separation of any increase in contaminants produced during the testing from the background level.

The recording instrument did not detect any change in the contaminant levels during tests 2 through 3f that was above that determined for the ambient air in test 1. Thus, the gaseous phase unburned hydrocarbon level in the bleed air remained the same as that in the ambient air during the entire time oil was being injected into the inlet. The validity of the data was shown by the positive level detected in the background and by the positive displacement that occurred when a gaseous hydrocarbon propane (C_3H_8) was injected into the engine inlet as a check of system response.

The glass wool liquid/vapor separation filter was removed after test 3(g) was completed. After the separation filter was removed, the flame ionization detector (FID) lines became contaminated with liquid oil as soon as the oil injection began, as indicated by the rise in unburned hydrocarbon level that occurred as soon as the oil was injected. The following observations show that the reading was produced by liquid contamination.

1. No increase in any other parameter was detected. A CO increase would be expected if the levels of unburned hydrocarbons were actually the result of oil breakdown.
2. The unburned hydrocarbon reading could be changed by changing the temperature of the sample line. This was a result of a change in the liquid's vapor pressure as the line temperature changed.
3. The indication continued after the oil flow had stopped, taking 10 minutes to decrease from an indication of 56 ppm. Since the indication continued after the oil flow was cut-off, the only source was material collected in the sample lines.

During test 4, the scale for unburned hydrocarbons was expanded 10 times so that each vertical division was 0.1 ppm and full scale was 10 ppm. This assured the detection of any liquid or gaseous substances that might be in the bleed air as a result of operating the engine with the carbon seal removed. A solenoid was installed in the sample line so that it was possible to switch between the engine bleed air line and the propeller airflow line to compare the bleed air with the ambient air conditions. Both the ambient air (inlet) and the bleed air remained at 2 ppm or less throughout the test and no difference could be noted between the bleed air and the ambient air contaminants. Since this test was run without the glass wool liquid/air separation filter, any leakage of oil into the bleed air system, whether, as a liquid or gaseous constituent, should have been detectable.

The engine was shutdown for 1 hour to collect and measure the amount of oil that would pass into the inlet once scavenging was stopped. The amount of oil that was collected was 20 cubic centimeters (cc) over a 60-minute period. This indicated that with no mechanical seal present (or a cracked carbon seal) the condition would be detected by oil leaking out the inlet onto the ground.

Before the start of test 5b, a residual value of 20 to 30 ppm unburned hydrocarbons was detected due to a minute quantity of fuel collecting in the unventilated combustor section of the engine. During the start, the effect of the fuel vapors could not be distinguished from oil contamination of the bleed air resulting from the start with a "dirty" inlet. Within 1 1/2 minutes, the bleed air contaminant level had reduced to 4 ppm. Therefore, even with an accumulation of oil in the engine inlet, only a very short duration of low level bleed air contamination would occur.

The validity of the above data was demonstrated by the fact that the Garrett test equipment was calibrated before and after each test run with certified laboratory standard gases. After each test, the subsequent calibration showed that in no case had any change in the equipment calibration occurred. In addition, throughout the testing, propane injected at the inlet was readily detected by the FID equipment showing the validity of the test system for detecting unburned hydrocarbons.

The validity of the analysis for the unchanged liquid state of the oil is shown as follows:

1. When oil or propane was injected at the inlet, the contaminants were detectable in the bleed air.
2. When the liquid contaminant was separated from the bleed air during the oil injection tests, no gaseous contaminants were detected.
3. Since no gaseous contaminants were present but liquid contaminants were detected, then the oil was unchanged as it passed through the engine's compressor.

Additionally, the test conditions were such that they were more severe than would normally be encountered.

During the oil injection test, the outside air temperature (OAT) ranged from 110° F to 115° F. The effect on engine operation at this high temperature is an increase in engine compressor discharge temperature and a decrease in compressor discharge pressure. Both conditions will tend to encourage thermal breakdown of the oil injected into the engine.

Clayton Environmental Consultants Inc.

Gas Chromatography/Mass Spectroscopy Analyses.--The primary emphasis of Clayton's sampling and analytical program involved qualitative scans to identify potential contaminants present in the bleed air samples using Gas Chromatography/Mass Spectroscopy, GC/MS, followed by quantitative analysis via GC. Separate sampling was conducted for different engine-operating conditions which varied from normal to simulated problem situations. The samples taken using Tenax in series with activated carbon were intended for the qualitative analyses.

All of the Tenax-activated carbon series samples were drawn from the first port following the glass manifold intake. The same calibrated pump was used to control sampling flow rate. Manifold pressures as indicated by the monometer were maintained from 2 to 3 inches water gauge. Bleed air temperatures measured immediately downstream of the glass manifold were indicated ranging from 96° F to 107° F. This compares similarly with the ambient temperature experienced during the 2 days of tests.

Following sample collection, the separate Tenax and activated carbon sorbent tubes were removed from the glass manifold, sealed using polyethylene caps, and stored under refrigeration until analysis.

The GC/MS analyses were accomplished using a Hewlett Packard Model 5985 GC/MS equipped with a Century thermal desorber. This was followed by cryogenic focusing prior to sample injection. A 6-foot 3 percent SP 2100 column was utilized and temperature programmed from 40° C to 250° C at 8° C per minute. The sample was scanned from a molecular weight of 40 to 400 and identification of spectra was accomplished using the registry of mass spectra data. To verify the effectiveness of the analytical method, the blank samples were spiked with numerous compounds, including hexadecane, prior to analysis.

Table 12 (Appendix 6) shows that all of the compounds identified in the bleed air samples, other than trichlorotrifluoroethane, were also identified in the field blank, Sample TEN-J-blank. Additionally, the various alkyl benzenes suggested as present in samples TEN/ACT-TEN-II and TEN-J-I, are typically found in ambient air samples when using Tenax as the absorbing medium. Gas chromatography/mass spectroscopy analysis of the activated charcoal samples (taken in series, subsequent to the Tenax tubes) did not indicate the presence of any compounds.

The trichlorotrifluoroethane identified as present in sample TEN/ACT TEN-II from test 4 was probably a residue contaminant of the stainless steel sample line which had been rinsed with Freon 113 prior to test 4. No apparent explanation exists for the trichlorotrifluoroethane identified in sample TEN-J-I from test 3 other than possible contamination of the outdoor test cell air from Freon 113 used to clean oil from below the engine support stand.

A sample of the stainless steel sampling line rinse solution was concentrated via evaporation, and then analyzed by GC/MS. A sample of the ETO-2380 turbo oil from the supply, which was injected into the engine during test 3, was also subjected to GC/MS analysis using the same instrument conditions. The total ion current chromatograms produced during these two analyses were compared by the analyst and a similarity was noted with respect to specific peaks of comparable relative size having similar retention times. The mass spectra for some of the comparable peaks matched, thus indicating the presence of compounds with identical empirical formulae.

In summary, the absence of identifiable aliphatic and aromatic hydrocarbons, esters, ketones, and aldehydes from bleed air samples taken during oil injection tests (test 3) suggests that the oil and the additives did not undergo any detectable degradation as these lubricant components passed through the two stages of compression and out the bleed air port.

Oil Mist Analyses.--The data contained within table XIII (Appendix G), shows that the gravimetric analyses of the oil mist samples taken during oil injection tests (test 3 a-f) progressively increased in weight as the oil injection rate was increased. Further analyses using fluorescence techniques identified the majority of the materials trapped by the sampling filters as the principle lubricating ingredient in ETO-2380 turbo oil.

These results support those from GC/MS analyses regarding the lack of evidence of degradation oil as it passed through the engine.

Two of the filter samples taken during the oil injection tests were analyzed for tricresylphosphate, the antiwear additive in ETO-2380 turbo oil. These samples included AA-187-20 (6 lbs/hr oil injection) and AA-187-24 (12 lb/hr oil injection). Results of the analyses indicated no triparacresylphosphate (TPCP) or triorthoeresylphosphate (TOCP) in either sample at the limits of detection for the analytical method employed.

Exxon's ETO-2380 turbo oil is subject to MIL specification L-23699C which requires that the tricresylphosphate additive contains not more than 1 percent of the ortho isomer. This same specification allows antiwear additive concentrations up to 5 percent by weight, thus the maximum allowable TOCP concentration in the oil is 0.05 percent.

Quantitative Analyses.--Separate air samples were taken during each test specifically to allow quantitative determination of contaminants identified by GC/MS. Ambient air samples were also taken at the test cell fence line upwind of the engine's intake to allow exclusion of contaminants introduced via the ambient air.

Since no bleed air contaminants, other than trichlorotrifluoroethane, were identified during GC/MS analyses, the subject samples were all analyzed for total hydrocarbon content to assess the degree of overall organic chemical contamination. The results of these analyses, as presented in tables XIV (appendix G), are expressed in ppm calculated as n-hexane. The indicated total hydrocarbon concentrations for bleed air samples from all tests varied from less than 0.02 to 2.0 ppm as n-hexane. The current Occupational Safety and Health Administration (OSHA) permissible exposure level (PEL) for petroleum distillates is 500 ppm as n-hexane for 8-hour time weighed average (TWA) exposures, representing the allowable concentration to which workers may be exposed for up to 8 hours per day, 40 hours per week. The slightly higher total hydrocarbon concentrations indicated by sample ACT-12 for test 5 which was a "dirty" startup are the possible result of bleed air contamination from engine fuel vapors which apparently diffused into the engine's compressor section between engine shutdown after the pretest runup and the "dirty" start.

Comparison of total hydrocarbon concentrations for ambient air samples taken at the test cell fence line with the engine intake air samples from test 1 showed no significant differences. This supported the concept of fence line ambient air sampling as an indication of engine intake air quality.

Analyses for para(p)-cresol, and formaldehyde were also conducted on the bleed and ambient air samples. P-cresol is considered a possible breakdown product of any TCPCP antiwear additive in ETO-2380 turbo oil. The results of analyses failed to indicate the presence of p-cresol at the limits of detection for the sampling and analytical methods employed. Formaldehyde is a common oxidation product of organic compounds; the presence of which in bleed air at elevated concentrations could indicate possible degradation of engine-ingested organic chemical contaminants. The results of analyses for formaldehyde indicated similar concentrations in bleed and ambient air, ranging from less than detectable to 0.5 ppm, as TWAs. It is significant to note that no formaldehyde was detected in the bleed air during test 5, the "dirty" startup.

Exxon Research and Engineering Company Discussion of Test Data

Table XV (appendix G) presents the data that was obtained on the samples collected by the freeze out train. Since the sampling tube contained both a gaseous phase and a water phase the contaminants were measured for both phases. The tubes which correspond to the various tests are identified in table XVI (appendix G).

The ppb contaminants for each tube is the sum of the vapor phase plus the aqueous phase and are summarized in Table XVII.

Table XVII.--Summary of the sum of the vapor phase plus the aqueous phase.

Test No.	Front Tube		Rear Tube		Total ppb	pb
	Vapor Phase ppb	Aqueous Phase ppb	Vapor Phase ppb	Aqueous Phase ppb		
1	1.1	6.1	—	—	7.2	0.007
2	0.3	28.6	0.5	2.2	31.6	0.032
3	7.1	12.5	8.0	—	27.6	0.028
4	1.2	10.2	0.7	1.9	14.0	0.014
5	0.1*	2.8	Broken		2.8	0.003

*Less than.

It is readily seen that the chemical fragments or contaminants were present in such small quantities that it was virtually impossible to identify them. Under these circumstances, the concentrations were insufficient to get definitive mass spectrometric data to apply molecular weights to the peaks found on the gas chromatograms. Since two different compounds may have the same retention time when run under identical GC conditions, it is not possible to positively identify a compound based on its retention time. However, a given compound cannot have two different retention times for a given set of GC conditions. Therefore, it was possible to demonstrate that several compounds, including acrolein, benzene, toluene, and xylene, were not present in any of the samples.

Two persistent contaminants were isopropyl alcohol and Freon TF; the former was from the dry ice bath and the latter was used by Garrett for washing sampling lines and engine and hardware parts. The data contained in table XVII (appendix G) showed that these were the only compounds detected in the Tenax columns.

It was surprising that during test 3, the oil injection tests the FID did not detect any contaminant in the bleed air until the maximum rate of 12 lbs/hr was reached and the glass wool plug which was in the line to catch particles or mist was removed. Unfortunately, the glass wool was lost but the Freon rinse from the trap was analyzed and the gas chromatogram was that of ETO-2380 turbo oil. This indicates that only turbo oil was coming through in this maximum contamination exercise and no breakdown products were observed.

The results of the gas chromatographic analyses of the organic components in the glass dry ice/isopropanol (cryogenic) traps are presented in tables XV, XVI, and XVIII (appendix G). The total organic components found in the compressor bleed air and in the intake air to the engine are presented in table XVI (appendix G). The intake air contained approximately 0.007 parts per million (ppm) of total organic components. The total organic components in the compressor bleed air ranged from 0.003 to 0.032 ppm. Neither the addition of ETO-2830 turbo oil into the intake of the engine nor the removal of the carbon seal from the engine resulted in a significant increase in total organic components in the compressor bleed air.

The distribution of components between the vapor phases and the condensed aqueous phases in the cryogenic traps are presented in table, XV (appendix G). As would be expected, the condensed aqueous phases contained the higher organic component concentrations. Tube 7 contained insufficient aqueous phase for sampling and analysis.

The distribution of organic components in the vapor and aqueous phases based on retention times of the cryogenic traps are presented in table XIX. The vapor phases contained up to 11 components with retention times in the range of 0.84 to 6.60 minutes. The aqueous phases contained two to five components with retention times in the range of 0.86 to 3.02 minutes. One of the components with a retention time of 1.92 minutes in the vapor phase and at 1.63 minutes in the aqueous phases was tentatively identified as isopropanol based on retention time.

Since isopropanol was used in the coolant medium during sample collection, it was suspected that the cryogenic tubes were contaminated with this component during sampling. This concentration was, therefore, not included in the total concentration. The fact that components in the 3.37- to 6.60-minute retention time range were found in the vapor phase but not in the aqueous phase suggests that these components are less polar and are probably hydrocarbons containing no functionality.

Other components were found in the vapor phases of the cryogenic traps in the 8.9 to 19.4 minutes (8.9, 12.9, 16.1, and 19.4 minutes) retention time range. These components were identified by GC/MS as containing a: ion fragment (m/e) of 74 mass units indicative of low molecular weight (MW) silicone grease components. Indeed, a sample of the silicone grease, when placed in a separate vial and when the headspace was analyzed, contained these same component peaks. Since these were known grease components and their presence in the compressor bleed air could not be explained based on any of the components used in ETO-2380 turbo oil, they were not included in the total component distribution. The concentrations of the other components were too low to be characterized or identified further by GC/MS analysis.

Although the levels of hydrocarbons present in the cryogenic tube were too low to allow identification by GC/MS analysis, their levels were detectable by flame ionization and tentative identifications were made on the basis of GC retention times. It was possible to suggest that the peak at 0.86 minute was consistent with that of methanol. The other component peaks were indicative of C_5 to C_7 normal and branched paraffins or olefins, C_3 to C_5 alcohols, or C_3 to C_5 ketones. Based on retention times of known compounds injected into the GC under the same conditions as the samples, known components such as acrylonitrile, acrolein, benzene, toluene and xylenes were not present in any of these samples.

The results of the GC analysis of the organic components adsorbed in the Tenax tubes are presented in table XIX (appendix G). Two component peaks were detected in tubes 1 and 2. The level of these components in the intake and compressor bleed air were not significantly different. These peaks were tentatively identified on the basis of retention time as Freon TF or methanol and isopropanol. Tubes from runs 4 and 5b contained inordinately high levels of a single component peak. The peak's identification, as based on retention time, was Freon TF; Freon TF previously was used to wash out the sampling lines. No other components of significance were noted.

Environmental Protection Agency, Discussion of Test Data

Eight precleaned Tenax cartridges were used for this test program. Two of the eight cartridges were used as blanks (unexposed to the ambient air) and one cartridge was placed in series (backup) during test 3b to provide a measure of sampling efficiency.

When the sealed metal container which contained the sampling cartridges in the culture tubes was opened at the test site, all of the culture tubes were cracked and that some of the cartridge ends had been broken off; however, the cartridges were used for sampling, but were stored in pint glass bottles wrapped with aluminum foil for transport back to the EPA laboratory. The results indicated that some cartridge contamination had occurred to each sample.

The quantities of air collected for each test are presented in table XX.

Table XX.--Summary of test results (EPA).

	<u>Sampling Time</u>	<u>Total Collected Sample (Liters)</u>
Test 1	1 hour	60
Test 2	1.5 hours	90
Test 3g (A) *	15 minutes	15
Test 3g (~) **	15 minutes	15
Test 4	1.5 hours	90
Test 5 (b)	10 minutes	10

*Normal sample cartridge used during test 3g.

**Backup sample cartridge used during test 3g.

A list of the compounds tentatively identified in each sample are presented in tables XXI through XXVIII (Appendix G). The identifications are only tentative in that standards were not used for the confirmation of these compounds. Although a high resolution capillary column was used for the analyses of these samples, overlapping of peaks was prevalent due to the large number of compounds present. In many cases (tent.) was added after the compound identified to indicate that this identification was a best estimate of the structural formula. The total ion abundance for each compound which is measured by the detector of the mass spectrometer and directly related to peak area was also listed; but, no quantitative results were obtained since standard calibrations had not been performed. The sensitivity of the mass spectrometer detector was increased approximately 2 1/2 times for the analyses of tests 2, 3gB, and 5b as compared to the blank and other test samples.

All of the sampling cartridges, including Blank #1, were found to contain many of the compounds associated with the analyses of diluted automobile exhaust. These results appeared to confirm the contamination of the cartridges during transport. There were, however, significant differences between the samples in the types of compounds identified that were not related to this contamination. These differences are described briefly below:

Test 1: Background

The major constituents of this sample consisted of four branched chain alkanes (spectrum #'s 29, 32, 38, 41) which were probably C_9 isomers since these compounds occurred in the chromatogram at retention times between n-octane (C_8H_{18}) and n-nonane (C_9H_{20}), and the fact that this column specifically separates hydrocarbons based on their boiling point differences.

Test 2: Regular Run, Good Seal

It appeared that this cartridge could have been contaminated with a hydrocarbon lubricating oil and/or fuel, since more than 100 peaks (compounds) were obtained from this sample. All of the components in this sample (especially the hydrocarbons) were at relatively higher concentrations than any of the other samples analyzed in this study. The 2-methyl pentanol and n-butyl acetate identified in this sample are usually found as contaminants in sampling lines if tygon or plastic tubing are used in the sampling.

Test 3gA: 12 lbs/hr oil passed through the engine.

Approximately 25 percent of this sample consisted of a number of oxygenated compounds, (peaks 13, 23, 24, 30, 32, 41, 44 and 46). These compounds could have been from the oxidation of the lubricating oil as the oil was passed through the hot engine.

Test 3gB: Backup Cartridge

The results of this test indicated that breakthrough of some of the compounds collected in test 3gA had occurred as most of the same compounds (including the oxygenates) identified in test 3gA were also identified in the sample collected for test 3gB. The appearance of the hydrocarbons (toluene and n-octane for example) in both samples may be due to the contamination of the cartridges rather than breakthrough of these compounds through the Tenax.

Test #4: Regular Run. Oil seal removed.

Except for the unidentified compounds with the suspected structural formulas C_5H_{10} (spectrum #12) and C_6H_{12} (spectrum #16), this sample appears to contain many of the compounds that are present (toluene, n-octane, xylenes) when diluted automobile exhaust is collected and analyzed using these same Tenax cartridges.

Test 5b: Hot Start

The results obtained for this sample are similar to that obtained for test 4. The compounds identified are at relatively low concentrations as compared to test 4 mainly because the sampling time was only 10 minutes for this test vs. 1.5 hours sampling time for test 4.

Evaluation of Test Results

In considering the EPA data, it is apparent that the testing was conducted in such a way as to provide qualitative results rather than quantitative concentrations of possible airborne contaminants. In the opinion of the committee, any study intended to demonstrate possible toxic effects on humans by airborne contaminants should provide an indication of the concentrations for comparison against published toxicological

information. The methods used by EPA are of primary usefulness in determining trace organic vapor pollutants in ambient atmospheres. Though the committee is not aware of the actual limits of detection which these sampling and analytical techniques provide, review of pertinent literature indicates a high degree of sensitivity to very low pollutant concentrations. It is apparent that toxic effects to aircraft crewmembers would only result from breathing air contamination of sufficient concentrations to cause acute effects. For most substances, there are several orders of magnitude separating trace and acute symptom causing concentrations.

Published exposure limits, which include OSHA permissible exposure limits (PELs), National Institute for Occupational Safety and Health (NIOSH) recommended exposure limits and the American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit values (TLVs), are primarily intended to protect workers against chronic health effects. Where available, published exposure limits are based on 8-hour exposure periods; these limits are generally lower than the acute effect concentrations and are several orders of magnitude higher than trace concentrations. Thus, although the EPA analyses suggest the presence of a number of compounds in the bleed air under various conditions of the test protocol, the concentration of these substances appears to be at such a low level as to cause no acute degradation of pilot performance.

A further complicating factor in interpreting the EPA data was the fact that breakage of the pre-cleaned media sample containers (culture tubes) had occurred prior to sampling, thus necessitating use of pint glass bottles for sample isolation. Due to the breakage, all of the media, including the two blank cartridges were exposed to the ambient air at the test site. No discussion was provided regarding the source or nature of the pint glass bottles used for shipment to the laboratory. Thus, any possible sample contamination introduced by these bottles cannot be determined.

Tables XXI through XXVIII show the results of analyses reported by the EPA for blanks 1 and 2, and tests 1, 2, 3A and B, 4, and 5. For each test, a review was conducted for the list of compounds identified and in the column labeled Possible Contaminant, all instances were noted where any compounds were also identified as present in either of the blank samples for test 1 (background) or the blank and/or background samples for tests 2 through 5. The committee noted that of the 46 compounds identified in the background, 14 of these compounds also appeared in the blanks. For test 2 (good seal) of 41 compounds identified, all but 8 of these appeared in the blanks and/or the background. Of these 8, 5 are listed as isomers of C_8H_{16} . For test 3gA (12 lbs/hr oil ingestion), of 32 compounds identified, all but 7 appeared in the blanks and/or the background. For test 3gB (backup to 3gA) of 25 compounds identified, all but 7 appeared in the blanks and/or background. For test 4 (oil seal removed), of 15 compounds identified, all but 2 appeared in the blanks and/or background. For test 5b (hot start), of 7 compounds identified, all were present in the blanks and/or background.

The above comparisons illustrate a considerable degree of sample contamination. If these EPA data are accurate, the number and type of compounds indicated as present in the ambient air is significant. With the degree of sample contamination indicated by these data, it is questionable whether or not any of these samples provides a true representation of actual compounds present in the air being studied.

The significance of the total abundance numbers which are included in the EPA's data are that these numbers are an indication of the mass spectrometer detector's degree of response for each compound identified. This response is a function of several variables,

including instrument conditions, compound type, and abundance. These numbers, while affected by the quantity of material present at the detector, cannot be used to determine that quantity unless known quantities of identical standards are run through the instrument under identical conditions. Thus, no estimates of compound abundance can be made on these data. In their discussion of the test results, the EPA noted that the mass spectrometer detector sensitivity was adjusted during the course of analyses. This adjustment precludes comparison of total ion abundance values from test to test. Since the instrumentation sensitivity used by EPA was indicated to be very high, their analyses identified many compounds which were probably in the trace concentration range. Most of these were found in the blank/background samples and, therefore, showed that contamination did occur.

APPENDIX G

ENGINEERING TABLES

Table XII.--Summary of gas chromatography/Mass spectroscopy analysis

Sample ID	Type	Test Description	Analytical Findings
TEN-J-blank	600 mg TENAX	Blank Sorbent Tube	$C_{10}H_{12}$ possible ethyl styrene Naphthalene 4 each probable hydrocarbons
TEN/ACT -J TEN-1	600 mg TENAX	Primary sorbent tube run in series with 600 mg ACT during 120/hour oil injection, Test Three	Naphthalene $C_{10}H_{12}$ possible Dimethyl styrene
TEN/ACT -J ACT-1	600 mg ACT	Secondary sorbent tube run in series behind 600 mg TENAX during 120/hour oil injection, Test Three	No compounds identified
TEN-J-1	600 mg TENAX	Individual sorbent tube run parallel with above (2) during 120/hour oil injection, Test Three	$C_{10}H_{14}$ alkyl benzene $C_{10}H_{12}$ possible ethyl styrene $C_{10}H_{12}$ possible dimethyl styrene Naphthalene $C_{11}H_{14}$ alkyl benzene trichloro trifluoro ethane
TEN/ACT TEN-11	150 mg TENAX	Primary sorbent tube run in series with 150 mg ACT during engine test without carbon seal, Test Four	$C_{10}H_{12}$ probable alkyl benzene Naphthalene $C_{10}H_{12}$ possible ethyl styrene trichloro trifluoro ethane
TEN/ACT ACT-11	150 mg ACT	Secondary sorbent tube run in series with above during engine test without carbon seal, Test Four	No compounds identified
TEN/ACT TEN-12	150 mg TENAX	Primary sorbent tube run in series with 150 mg ACT during dirty startup, Test Five	Trichloro trifluoro ethane $C_{10}H_{12}$ possible ethyl styrene $C_{10}H_{12}$ possible dimethyl styrene Naphthalene
TEN/ACT ACT-12	150 mg ACT	Secondary sorbent tube run in series with above during dirty startup, Test Five	No compounds identified
Freon Wash	Liquid bulk Freon 113	Sample of line wash solvent following oil injection tests, note sample concentrated 20:1	No compounds identified however, total ion current chromatogram resembles that of Exxon 2390 and some mass spectra match
Lab Blank	150 mg TENAX	Blank sorbent tube	No compounds identified

Table XIII.--Comparison of oil injection rates versus identified materials.

Date	Sample Number	Test Conditions	Sampling Start	Period Stop	Weight Gain (mg)	Oil Mist Exxon 2380 (mg)	Tri pure cresyl phosphate (mg)	Tri is the cresyl phosphate (mg)
7/21/81	AA-187-18	Oil Injection at 20/hr	1535	1542	0.35	0.24	-	-
7/21/81	AA-187-21	Oil Injection at 40/hr	1544	1549	0.46	0.65	-	-
7/21/81	AA-187-20	Oil Injection at 60/hr	1551	1556	2.01	-	< 0.030	< 0.030
7/21/81	AA-187-17	Oil Injection at 80/hr	1557	1602	3.17	3.1	-	-
7/21/81	AA-187-24	Oil Injection at 120/hr	1605	1610	4.10	-	< 0.030	< 0.030
7/21/81	AA-187-23	Oil Injection at 120/hr	1611	1616	5.51	5.2	-	-

Table XIV.--Total hydrocarbon content versus degree of overall organic contamination.
(Material(s) total hydrocarbons as n-hexane (THC), p-cresol

Date 1981	Sample Number	Description	Sampling Period		Sample Volume (liters)	Concentration		
			Start	Stop		THC (ppm)	P-cresol (ppm)	MCHO (ppm)
7/21	ACT-1	TEST ONE	1100	1200	7.9	<0.04		
7/21	TEN-1	Engine Intake Air	1100	1200	8.2		<0.03	
7/21	FORM-1		1100	1200	60			<0.01
7/21	ACT-2	Fenceline Ambient Air	1013	1220	15.9	0.02		
7/21	TEN-2	Relating to Test One	1013	1218	14.8		<0.02	
7/21	FORM-2		1013	1225	150			0.005
7/21	ACT-3	TEST TWO	1240	1410	12.0	<0.02		
7/21	TEN-3	Normal Engine	1240	1410	12.4		<0.02	
7/21	FORM-3		1240	1410	90			0.009
7/21	ACT-4	Fenceline Ambient Air	1250	1725	34.5	0.06		
7/21	TEN-4	Relating to Tests Two and Three	1250	1725	32.5		<0.07	
7/21	FORM-4		1250	1725	314			0.007
7/21	ACT-J-1	TEST THREE (g)	1647	1702	2.0	<0.03	<0.1	
7/21	FORM-5	12#/hour oil inject	1647	1702	15			0.5
7/22	ACT-10	Fenceline Ambient Air	0830	1340	18.9	0.007		
7/22	TEN-10	Relating to Tests Four and Five	0800	1340	36.6		<0.006	
7/22	FORM-6							
7/22	ACT-11	TEST FOUR	0936	1104	11.6	0.3		
7/22	TEN-11	Engine running without carbon seal	0936	1104	11.9		<0.02	
7/22	ACT-12	TEST FIVE	1317	1327	1.3	2.		
7/22	TEN-12	Engine without carbon seal with "dirty" startup	1317	1327	1.4		<0.2	
7/22	FORM-12		1317	1327	10			<0.07

Table XV.--Distribution between vapor and aqueous phases of total organic components which were collected in glass dry ice/isopropanol traps at the Garrett Turbine Engine Company.

Tube No.	Sample Weight, g	Air Sampled, Liters (a)	Concentration in Sampled Atmosphere µg/L or ppb		
			Vapor Phase (b)	Aqueous Phase (c)	Total
1	2.058	158.4	1.1	6.1	7.2
3	4.556	374.9	0.3	28.6	28.9
4	0.304	374.9	0.5	2.2	2.7
6	0.464	65.3	7.1	12.5	19.6
7	0.066	65.3	8.0	(d)	8.0
8	3.420	368.8	1.2	10.2	11.4
9	0.600	368.8	0.7	1.9	2.6
10	0.388	44.6	-	2.8	2.8

(a) Total volume of air in liters corrected to 25°C and 760 mm Hg pressure.

(b) Total organic components in the compressor air in parts per billion (ppb) collected as a vapor based on a cryogenic tube volume of 95 mL.

(c) Concentration of components in the compressor air in parts per billion (ppb) collected in the cryogenic tube as in the condensed aqueous phase assuming an average molecular weight of components of 78.

(d) Insufficient aqueous phase for sampling and analysis.

Table XVI.--Total organic components collected in glass dry ice/isopropanol traps of the Garrett Turbine Engine Company.

Test No.	Tube No.	Tube Position	Sample Weight, g	Air Sampled, Liters (a)	Collection Time, Min.	Test Conditions	Total Organic Component µg/Liter or ppb
1	1	Front	2.058	158.4	60	Intake air - good seal - no oil injection - (Represents background air)	7.2
2	3	Front	4.556	374.9	90	Compressor bleed air - good seal - no oil injection	28.9
2	4	Rear	0.304	374.9	90	Compressor bleed air - good seal - no oil injection	2.7
3	6	Front	0.464	65.3	15	Compressor bleed air - good seal - oil injected into intake at 12#/hr.	19.6
3	7	Rear	0.066	65.3	15	Compressor bleed air - good seal - oil injected into intake at 12#/hr.	8.0
4	8	Front	3.420	368.8	90	Compressor bleed air - no carbon face on seal - no oil injection	11.4
4	9	Rear	0.600	368.8	90	Compressor bleed air - no carbon face on seal - no oil injection	2.6
6	10	Front	0.388	44.6	10	Compressor bleed air - no carbon face on seal - during start-up with oil pooled in intake	2.8

(a) Total volume of air in liters corrected to 25°C and 760 mm Hg pressure.

Table XVIII.--Total organic components collected in Texax traps at the Garrett Turbine Engine Company.

Texax Tube Test No.	Air Sampled, Liters ^(a)	Test Conditions	Total Organic Components, ppm, as			
			Freon ^(b)	(Methanol)	Isopropanol ^(c)	Total
1	4.0	Intake air - good carbon seal - no oil injection (represents background air)	75 (13)		45	120
2	4.0	Compressor bleed air - good carbon seal - no oil injection (represents normal engine operation)	75 (13)		63	138
3g	2.0	Compressor bleed air - good carbon seal - oil injected into intake at 120/hr.	—		—	(d)
4	3.8	Compressor bleed air - no carbon face on seal - no oil injection	3600 (600)		(e)	3600
5b	1.3	Compressor bleed air - no carbon face on seal - during start-up with oil pooled in intake	6400 (1100)		(e)	6400

(a) Total volume of air in liters corrected to 25°C and 760 mm Hg pressure.

(b) Freon (trifluorotrchloroethane) was used to wash sampling lines. This peak was found at the retention time of Freon and/or methanol.

(c) A peak formed at the retention time of isopropanol.

(d) This sample was used for the qualitative GC-MS analysis.

(e) No peak could be detected due to the unusually large concentration of a component eluting at the retention time of Freon.

Table XIX.--Distribution of individual organic compounds in the vapor and aqueous phase which were collected in glass dry ice/isopropanol traps at the Garrett Turbine Engine Company.

		Concentration, ppm, Present in							
Retention Time, Min.	File No.	Tube 1 LK193	Tube 3 LK739	Tube 4 LK740	Tube 6 LK671	Tube 7 LK715	Tube 8 LK679	Tube 9 LK686	Tube 10 LK705
<u>VAPOR PHASE(c)</u>									
0.84		-	-	-	-	5.0	0.8	1.6	-
1.61		-	0.1	0.6	0.7	-	0.8	-	-
1.92(a)		0.3(a)	0.7(a)	0.6(a)	0.3(a)	3.7(a)	1.3(a)	1.1(a)	1.2(a)
2.45		0.2	-	-	0.6	-	0.2	-	-
3.06		0.2	0.1	0.2	1.0	<0.1	0.5	0.2	-
3.37		0.2	<0.1	0.1	1.5	<0.1	0.3	0.1	-
3.74		0.5	0.3	0.4	0.3	0.1	0.6	0.3	-
4.33		0.1	-	-	-	0.1	0.3	0.1	-
5.13		0.5	0.4	0.5	0.5	0.1	0.6	0.3	-
6.08		0.1	0.1	0.1	0.3	-	0.4	-	-
6.60		-	-	0.1	-	-	0.1	-	-
TOTAL		1.8	1.1	2.0	4.8	5.3	4.6	2.6	-
<u>AQUEOUS PHASE(d)</u>									
	File No.	LK696	LK737	LK036	LK712	-	LK681	LK688	LK700
0.86		1.5	6.8	6.4	4.5	(b)	3.5	3.7	1.1
1.31		-	0.7	0.4	0.6	-	-	0.2	-
1.63		0.5(a)	1.2(a)	1.5(a)	3.8(a)	-	11.9(a)	2.7(a)	1.0(a)
2.65		-	-	2.0	0.1	-	-	-	-
3.02		-	-	-	0.4	-	-	-	-
TOTAL		1.5	7.5	8.6	5.6	-	3.5	3.9	1.1

(a) This peak has been identified as isopropanol based on retention time. Since isopropanol was used in the dry ice traps, this value is reported here but is not included in the total organic components.

(b) Insufficient aqueous phase was available for sampling.

(c) Concentrations in the vapor phase for a 3.0 cc sample size were calculated on the basis of a 4.6 ppm benzene blend which exhibited a response of 10.67 area units per ppm.

(d) Concentrations in the aqueous phase for a 2.0 µL sample size were calculated on the basis of a 4.6 µg/mL concentration of benzene in water which exhibited a response of 3.35 area units for 1.0 µg/mL concentration.

Tables XXI through XVIII.--EPA identified compounds and comparisons.

Table XXI

Blank #1

Spectrum #	Retention Time	Total Ion Abundance	Compound
2-5 ¹	16.8-17.3	-3	CCl ₃ F
6	17.8	3434	isopentane
7-8 ¹	17.8-18.0	-3	C ₂ Cl ₃ F ₃
13-16 ²	20.0-20.8	-3	CH ₂ Cl ₂
17	22.6	1606	C ₆ H ₁₄
18-20 ¹	24.0-24.3	-3	1,1,1 trichloroethylene
21	25.2	2886	benzene
22	26.9	1021	unknown
23	27.3	1127	C ₇ H ₁₆ (isomer)
24	27.4	1609	C ₇ H ₁₆ (isomer)
25	27.9	1658	C ₇ H ₁₆ (isomer)
26	29.4	1390	C ₂ H ₅ OH
27	29.8	6800	C ₃ H ₇ OH
28	31.7	3913	toluene
29	33.2	1029	C ₈ H ₁₈ (isomer)
30	34.4	1906	n-octane
31	38.0	3896	butanol
32 ²	-	-	-
33	44.4	1282	benzaldehyde

¹ G.C. column overloaded
² Mass spectrum uninterpretable
³ Mass spectrometer detector saturated

Table XXII (Pages 1 of 5)

Blank #2

Spectrum #	Retention Time	Total Ion Abundance	Compound
3-4	17.2-17.3	-3	CCl ₃ F
5-8	17.6-18.0	-3	isopentane
9	18.7	6978	C ₃ H ₁₀
10	19.8	2352	C ₂ Cl ₃ F ₃
11-15	19.9-23.3	-3	CH ₂ Cl ₂
16	31.6	1565	toluene

³ Mass spectrometer detector saturated

Table XXII (Pages 2 of 5)

Test #1 - Background

Spectrum #	Retention Time	Total Ion Abundance	Compound	Possible Contaminant
1-3	-	-	air	
4	17.1	1246	CCl ₃ F	blank
5	17.9	519	n-pentane	
6-7 ²	19.6-19.8	--3	C ₂ Cl ₃ F ₃	blank
8 ²	22.4	--	--	
9-11 ²	24.2-24.3	774	C ₇ H ₁₆ (isomer)(tent.)	
12	27.1	774	C ₇ H ₁₆ (isomer)(tent.)	
13	27.3	831	C ₇ H ₁₆ (isomer)(tent.)	blank
14	27.8	1592	C ₇ H ₁₆ (isomer)	blank
15	28.6	1736	C ₇ H ₁₆ (isomer)	blank
16	29.6	1238	C ₇ H ₁₆ (isomer)	blank
17	31.1	1155	propanol = C ₇ H ₁₆ (isomer)(tent.)	
18	31.3	693	C ₂ Cl ₄	
19	31.4	1769	unknown	
20	31.4	3491	toluene	blank
21	31.7	7490	C ₈ H ₁₈ (isomer)	blank
22	31.8	8482	C ₈ H ₁₈ (isomer)(tent.)	blank
23	32.3	3778	C ₈ H ₁₈ (isomer)	blank
24	32.5	1049	unknown	
25	32.8	791	C ₈ H ₁₈ (tent.)	blank
26	32.9	1536	C ₈ H ₁₈	blank
27	-	-	-	
28	34.1	6044	n-octane	blank
29	34.6	18308	C ₉ H ₂₀ (isomer)	
30	35.3	2056	C ₉ H ₂₀ (isomer)	
31	35.4	2023	C ₉ H ₂₀ (isomer)	
32	35.6	15622	C ₉ H ₂₀ (isomer)	
33	-	-	-	
34	35.9	6153	C ₉ H ₂₀ (isomer)	
35	36.1	1776	C ₉ H ₂₀ (isomer) = ethylbenzene	
36	36.2	1213	C ₉ H ₂₀ (isomer)	
37	36.2	3736	C ₉ H ₁₈ (isomer)	
38	36.3	15225	C ₉ H ₂₀ (isomer)	
39	36.5	1476	p-xylene - unknown	
40	36.6	2595	m-xylene	
41	36.7	14918	C ₉ H ₂₀ (isomer)	
42	37.0	1902	C ₉ H ₁₈ (isomer)	
43	37.2	3047	C ₉ H ₂₀ (isomer)	
44	37.6	907	unknown	
45	37.7	1907	styrene	
46	37.8	1937	C ₉ H ₂₀ (isomer)	
47	37.9	7222	n-nonane	
48	38.0	808	C ₁₀ H ₂₂ (isomer)	
49	38.2	3211	C ₁₀ H ₂₂ (isomer)	
50	38.4	1397	C ₁₀ H ₂₂ (isomer)	
51	38.9	704	C ₁₀ H ₂₂ (isomer) (tent.)	
52	39.0	1140	C ₁₀ H ₂₂ (isomer)	
53 ²	-	-	-	
54 ²	-	-	-	
55 ²	-	-	-	
56	41.1	822	C ₉ H ₁₂ (isomer)	
57	41.5	1338	C ₁₀ H ₂₂ (isomer)	
58	43.6	866	benzaldehyde	blank

- 1 G.C. column overloaded
 2 Mass spectrum uninterpretable
 3 Mass spectrometer detector saturated

Table XXII (Pages 3 of 5)

Regular Run (good seal)

Spectrum #	Retention Time	Total Ion Abundance	Compound	Possible Contaminant
1 - 36 data storage used for previous analysis				
35 - 36	-	-	air	blank and background
37	16.6	1571	CCl ₃ F	blank and background
38	16.9	913	isopentane	blank
39 ²	-	-	-	-
40	19.4	1177	C ₂ Cl ₃ F ₃	blank and background
41	20.9	1067	CH ₂ Cl ₂	blank
42	22.3	642	C ₆ H ₁₄ (isomer)	blank
43	22.1	1257	C ₆ H ₁₄ (isomer)	blank
44 ²	-	-	-	-
45-47 ²	24.3-24.4	- ³	1,1,1 trichloroethylene	blank
48	25.1	1613	benzene	blank
49	26.3	1417	C ₇ H ₁₆ (isomer) (tent.)	blank
50 ²	-	-	-	-
51 ²	-	-	-	-
52	27.1	2007	C ₇ H ₁₆ (isomer) (tent.)	blank and background
53	27.3	1662	C ₇ H ₁₆ (isomer)	blank and background
54	27.7	2006	C ₇ H ₁₆ (isomer)	blank and background
55	27.8	4528	C ₇ H ₁₆ (isomer)	blank and background
56	28.1	1764	C ₇ H ₁₆ (isomer)	blank and background
57	28.4	2049	C ₇ H ₁₆ (isomer)	blank and background
58	28.6	6040	C ₇ H ₁₆ (isomer)	blank and background
59 ²	-	-	-	-
60 ²	-	-	-	-
61	31.2	4189	C ₂ Cl ₄	background
62 ²	-	-	-	-
63	31.4	23553	toluene	blank and background
64	32.1	1458	C ₈ H ₁₈ (isomer)	-
65	32.3	6324	C ₈ H ₁₈ (isomer)	-
66	32.4	2609	C ₈ H ₁₈ (isomer)	-
67	32.5	4179	C ₈ H ₁₈ (isomer)	-
68	32.6	2480	C ₈ H ₁₈ (isomer)	-
69 ¹	32.8	4786	C ₈ H ₁₈ (isomer)	blank and background
70	32.9	14058	C ₈ H ₁₈ (isomer)	blank and background
71	33.0	11807	C ₈ H ₁₈ (isomer)	-
72	33.9	4119	C ₈ H ₁₈ (isomer)	-
73	34.1	35820	n-octane	blank and background
74	34.2	1562	2-methyl pentanol (tent.)	-
75 ²	-	-	-	-
76	34.9	2081	n-butylacetate (tent.)	-
77	35.5	4160	C ₉ H ₂₀ (isomer)	background
78	35.6	1612	C ₉ H ₂₀ (isomer)	background
79	35.7	3827	C ₉ H ₂₀ (isomer)	background
80	35.8	1371	C ₉ H ₂₀ (isomer)	background
81	35.8	1345	C ₉ H ₂₀ (isomer)	background
82	35.9	18563	ethylbenzene	background
83	36.1	14187	C ₉ H ₂₀ (isomer)	background
84	36.2	4095	C ₉ H ₂₀ (isomer)	background
85	36.2	1354	C ₉ H ₂₀ (isomer)	background
86	36.3	18064	p-xylene	background
87	36.4	43438	m-xylene	background
88	36.5	4693	C ₉ H ₂₀ (isomer)	background
89	36.6	26421	C ₉ H ₂₀ (isomer)	background
90	36.7	26421	C ₉ H ₂₀ (isomer)	background
91	36.9	22564	C ₉ H ₂₀ (isomer)	background
92	37.0	7996	o-xylene	background
93 ²	-	-	-	background
94	37.6	9893	C ₉ H ₂₀ (isomer)	background
95	37.7	37770	n-nonane	background
96 ²	-	-	-	background
97	39.4	4166	C ₉ H ₂₂ (isomer)	background
98	39.5	8619	C ₁₀ H ₂₂ (isomer)	background
99	39.8	9199	C ₉ H ₂₂ (isomer)	background
100 ²	-	-	-	background

- ¹ G.C. column overloaded
² Mass spectrum uninterpretable
³ Mass spectrometer detector saturated

Table XXII (Pages 4 of 5)

Test 3gA - 22 lbs/hr of oil run through engine

Spectrum #	Retention Time	Total Ion Abundance	Compound	Possible Contaminant
1-2	-	-	air	blank and background
3-5 ¹	16.8-17.1	-3	CCl ₃ F	blank and background
6-8 ¹	17.5-17.8	-3	isopentane	blank
9	18.4	5060	C ₅ H ₁₀ (isomer)	blank
10	19.1	709	n-pentane	background
11-12 ¹	19.4-19.6	-3	C ₂ Cl ₃ F ₃	blank and background
13	19.8	2848	propanol	background
14-15 ¹	19.8-22.6	-3	CH ₂ Cl ₂	blank
20-22 ¹	23.9-24.1	-3	1,1,1 trichloroethylene	blank
23	24.4	2719	n-butanol	blank
24	35.6	3229	2-butanone	
25	26.8	1020	C ₇ H ₁₆ (isomer)	blank and background
26	26.9	1231	C ₇ H ₁₆ (isomer)	blank and background
27	27.5	1921	C ₇ H ₁₆ (isomer)	blank and background
28 ²	-	-	-	blank and background
29	28.3	2624	C ₇ H ₁₆ (isomer)(tent.)	blank and background
30	28.3	1606	C ₅ H ₈ O (tent.)	
31	28.9	899	C ₇ H ₁₆ (isomer)	blank and background
32	30.4	6312	2-pentanone	
33	31.1	4253	toluene	blank and background
34	32.1	971	unknown	
35 ²	-	-	-	
36	32.6	1764	C ₈ H ₁₈ (isomer)	blank
37 ²	-	-	-	
38	33.4	2320	C ₈ H ₁₈ (isomer)	
39 ²	-	-	-	
40	33.8	2332	n-octane	blank and background
41	34.4	1499	C ₈ H ₁₈ O (tent.)	
42	35.6	10880	C ₉ H ₂₀ (isomer)	background
43	35.9	2753	ethylbenzene	background
44	36.0	1403	ethylpentanate (tent.)	
45	36.5	1978	p-xylene	background
46	36.8	1334	butyric anhydride (tent.)	

Test 3g(B) 22 lbs/hr Oil Run Through Engine - Backup Cartridge

Spectrum #	Retention Time	Total Ion Abundance	Compound	Possible Contaminant
1-2	-	-	air	blank and background
3	16.7	1230	CCl ₃ F	blank and background
4 ²	-	-	-	
5-6 ¹	19.2-19.6	-3	C ₂ Cl ₃ F ₃	blank and background
7	20.3	931	propanol (tent.)	background
8 ²	-	-	-	
9-10 ²	21.9-22.0	1224	acetone	
11-12 ²	24.0	9574	1,1,1 trichloroethylene	blank
13	24.4	1655	n-butanol	
14	24.5	3140	n-butanol	blank
15	24.7	1348	benzene	blank
16	25.5	2474	2-butanone	
17	26.8	1346	C ₇ H ₁₆ (isomer)	blank and background
18	27.4	2168	C ₇ H ₁₆ (isomer)(tent.)	blank and background
19 ²	-	-	-	
20	28.3	1289	C ₅ H ₈ O (tent.)	
21	30.4	2948	n-pentanol (tent.)	
22 ²	-	-	-	
23	31.0	1894	toluene	blank and background
24-27	31.5-32.1	1534	ketone M.W. 170 (tent.)	
28	32.4	2255	C ₈ H ₁₈ (isomer)	blank and background
29	32.4	2255	C ₈ H ₁₈ (isomer)	blank and background
30	33.7	2832	n-octane	blank and background
31	35.4	5277	C ₉ H ₂₀	blank and background
32 ²	-	-	-	
33	35.8	2211	ethyl benzene	background
34	36.3	1540	p-xylene	background
35	37.0	1721	o-xylene	
36	37.4	2588	styrene	background
37	37.7	3021	C ₉ H ₂₀ (isomer)	background
38 ²	-	-	-	

¹ G.C. column overloaded² Mass spectrum uninterpretable

APPENDIX G

Table XXII (Pages 5 of 5)

APPENDIX G

Test #5 Oil Seal Removed

Spectrum #	Retention Time	Total Ion Abundance	Compound	Possible Contaminant
1-2	-	-	air	blank and background
3	18.9	2271	CCl_3F	blank and background
4-9 ¹	19.3-20.6	- ³	1,1,1 trichloroethylene	blank
12	28.8	941	C_5H_8 (tent.)	
13 ²	-	-	-	
14	31.4	2865	toluene	blank and background
15	34.1	2840	n-octane	blank and background
16	35.8	3242	C_8H_{10} (tent.)	
17	36.0	1935	ethylbenzene	background
18	36.5	1242	p-xylene	background
19	36.3	2458	m-xylene	background
20	37.0	1390	C_9H_{20} (isomer) = C_9H_{18} (tent.)	blank and background
21	37.1	1443	C_9H_{20} (isomer)	background
22	37.6	1759	styrene	background
23	37.8	1629	C_9H_{20} (isomer)	background

- ¹ G.C. column overloaded
² Mass spectrum uninterpretable
³ Mass spectrometer detector saturated

Test #5b - Hot Start

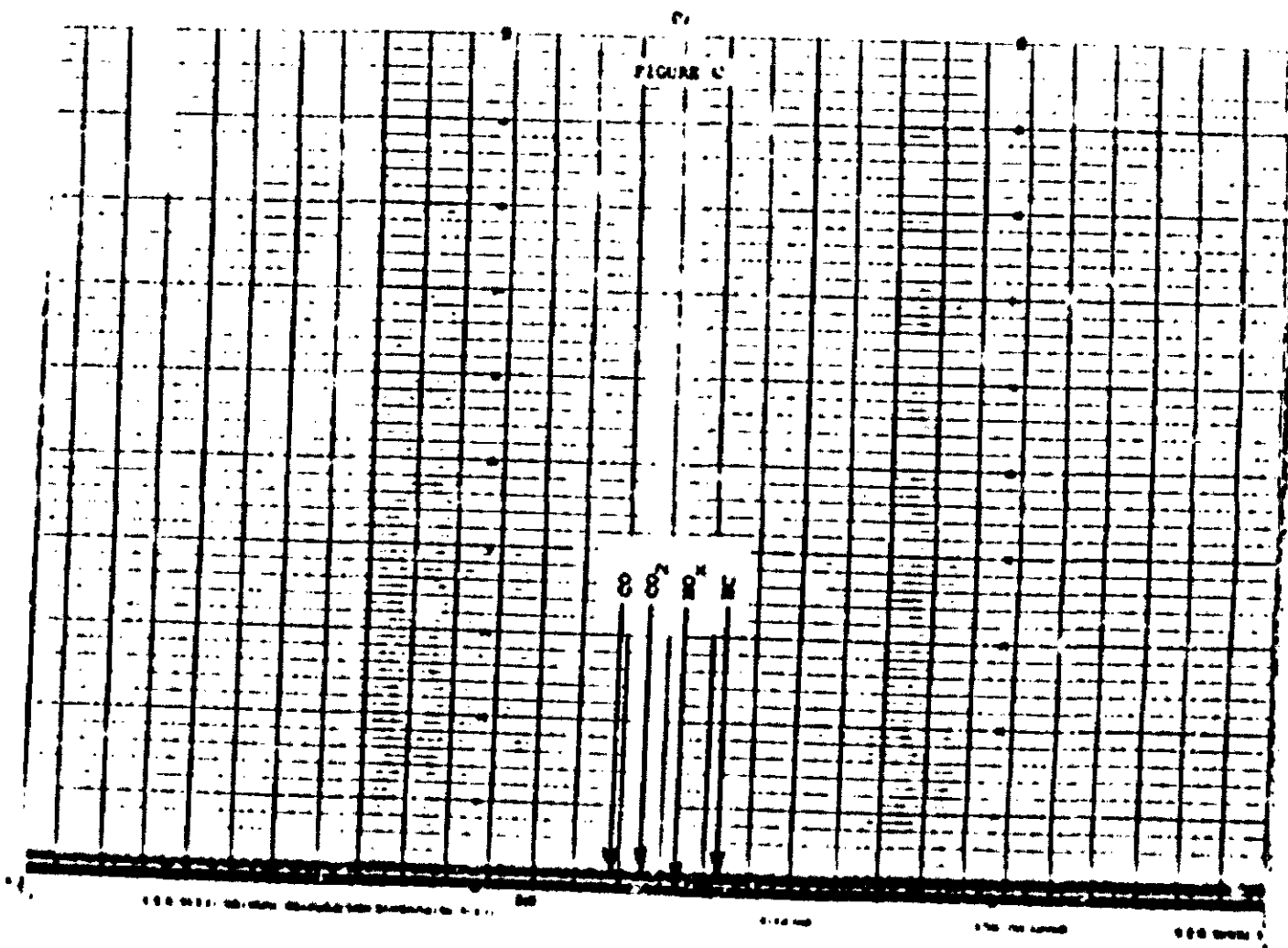
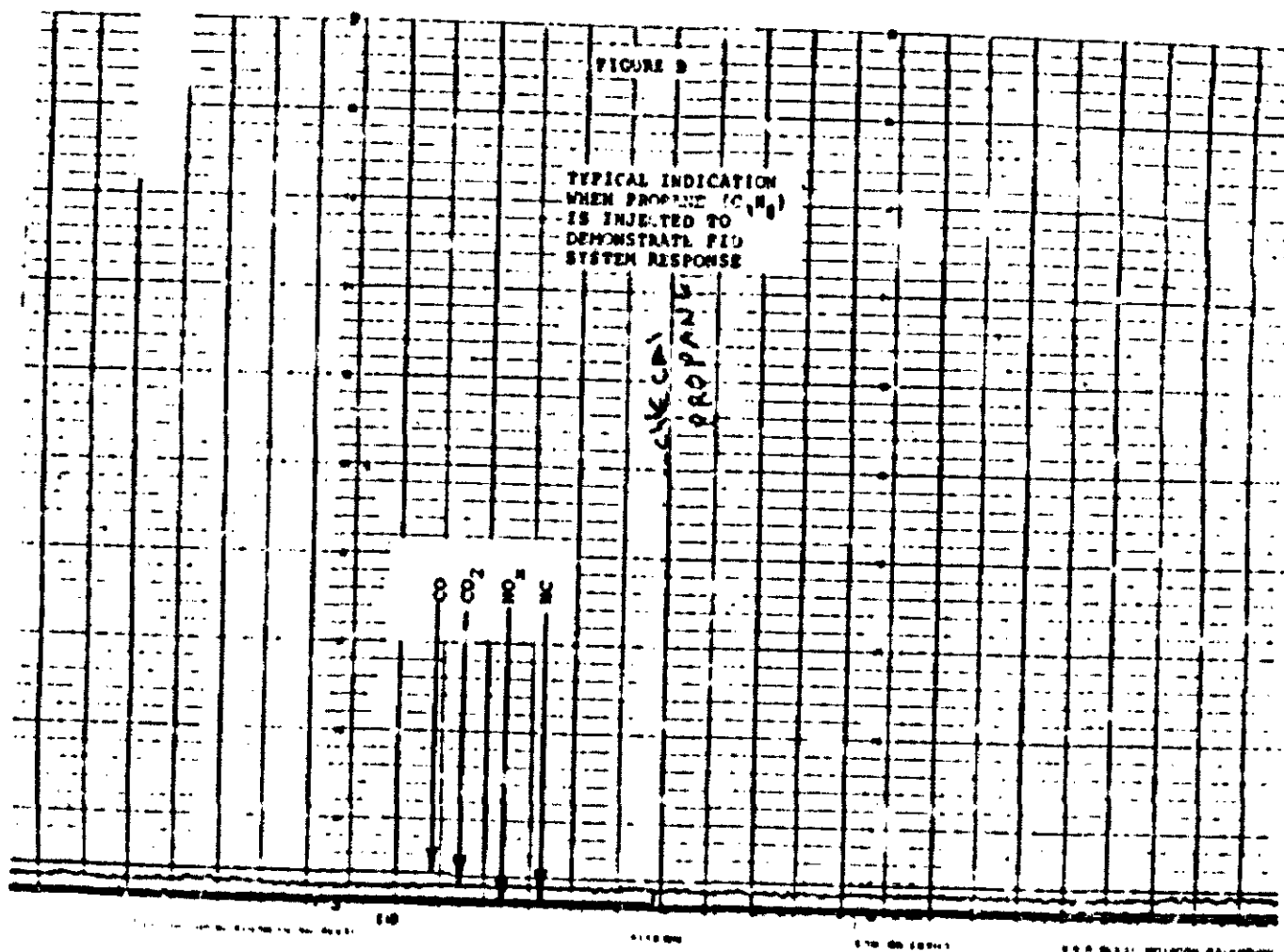
Spectrum #	Retention Time	Total Ion Abundance	Compound	Possible Contaminant
1-2	-	-	air	blank and background
3-9 ¹	19.1-19.9	- ³	$C_2Cl_3F_3$	blank and background
10 ²	-	-	-	
11 ²	-	-	-	
12-14 ¹	23.7-23.8	- ³	1,1,1 trichloroethylene	blank
15	24.5	1203	benzene	blank
16 ²	-	-	-	
17	27.9	-	C_7H_{16} (tent.)	blank and background
18 ²	-	-	-	
19	30.8	1102	C_2Cl_4	background
20	30.9	3348	toluene	blank and background
21-22 ²	-	-	-	
23	33.6	1516	n-octane	blank and background
24 ²	-	-	-	
25 ²	-	-	-	
26	35.6	1441	ethyl benzene	background
27	35.9	2191	C_9H_{20} (isomer)	background
28	36.1	1442	p-xylene	background
29	36.3	1639	m-xylene	background
30	36.7	2933	C_9H_{20} (isomer)	background
31	36.9	3172	C_9H_{20} (isomer)	background
32	37.4	2302	styrene	background
33	37.6	1827	C_9H_{18} (isomer)	background
34	37.6	6572	n-nonane	background

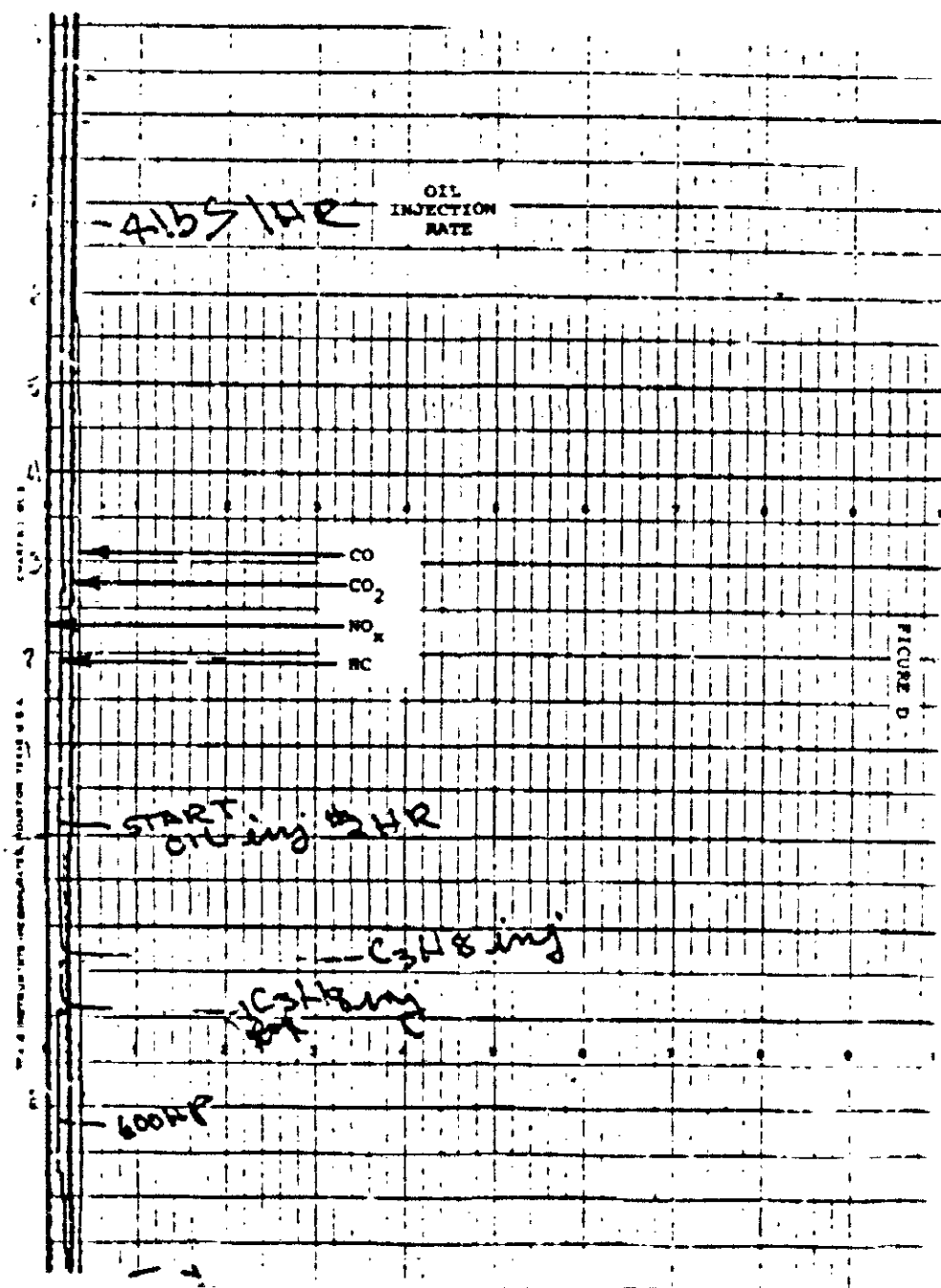
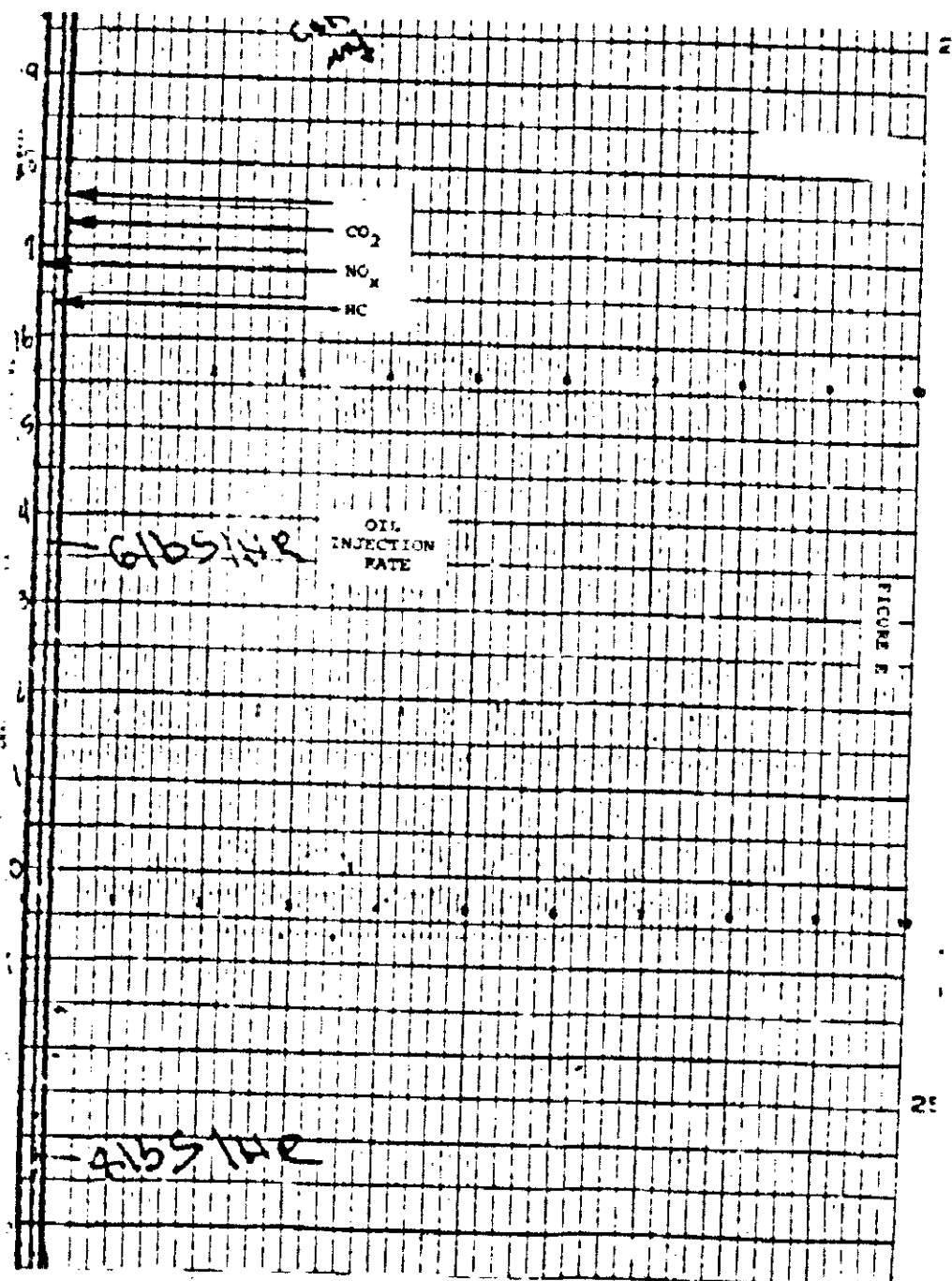
- ¹ G.C. column overloaded
² Mass spectrum uninterpretable
³ Mass spectrometer detector saturated

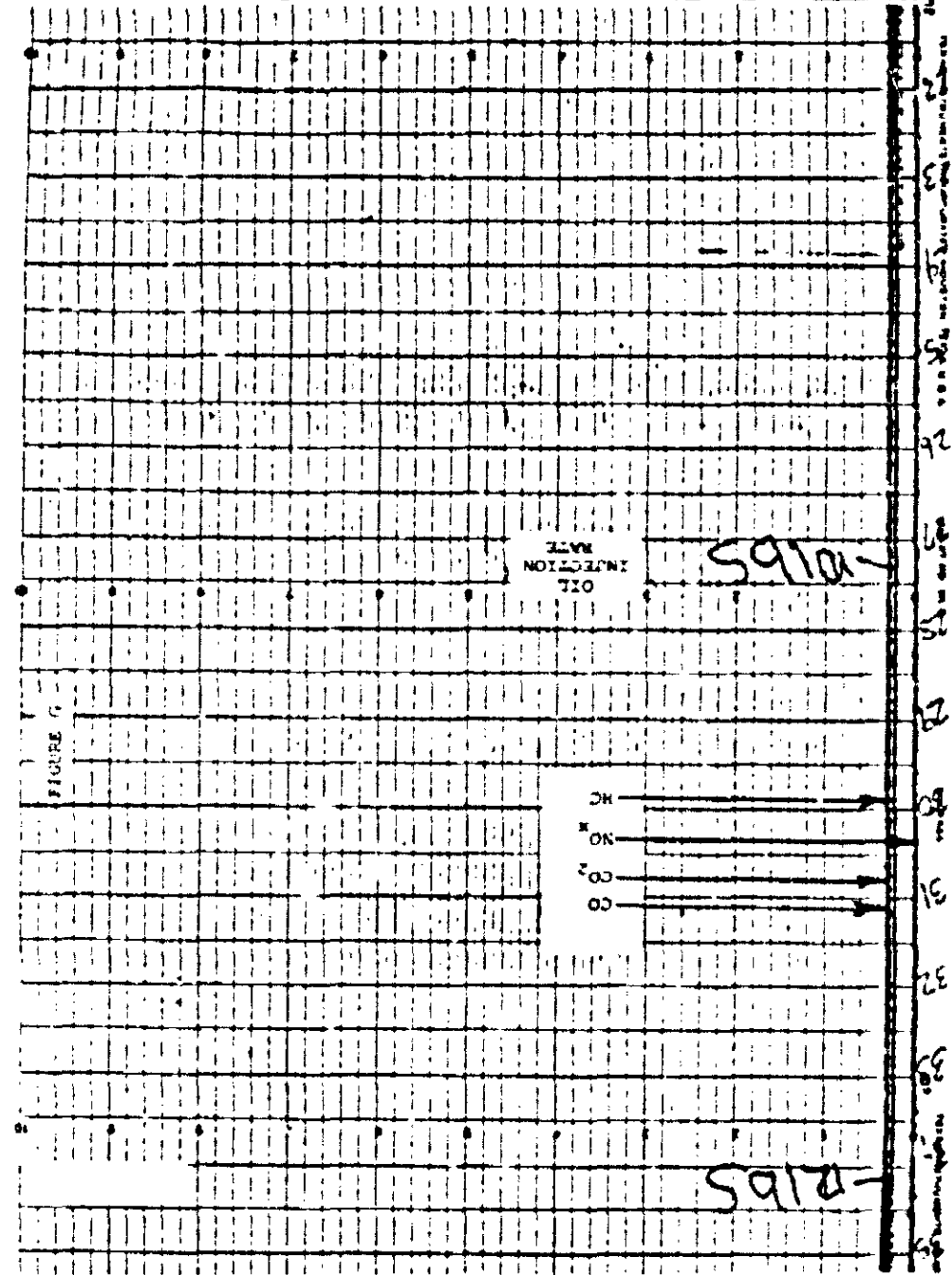
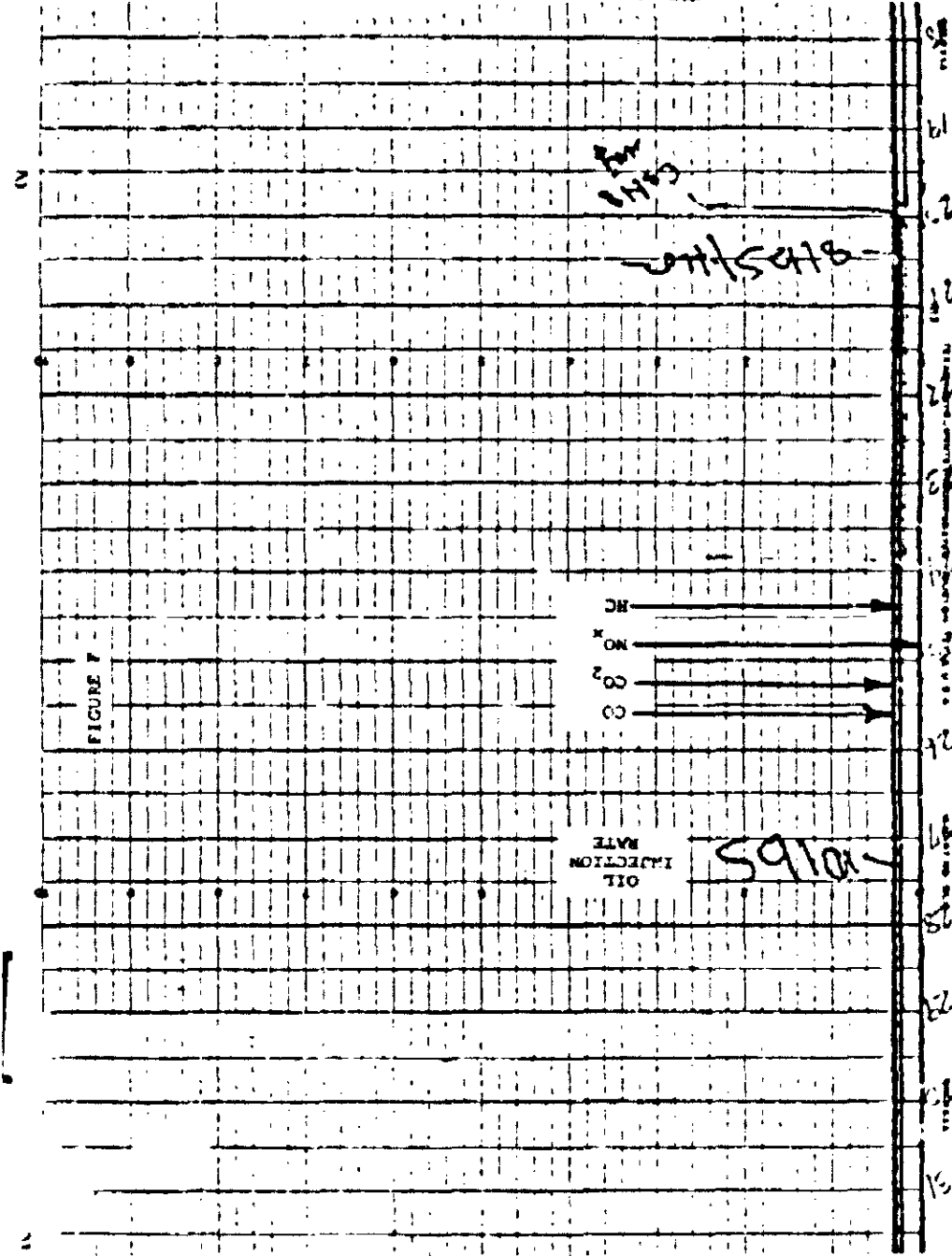
-76-

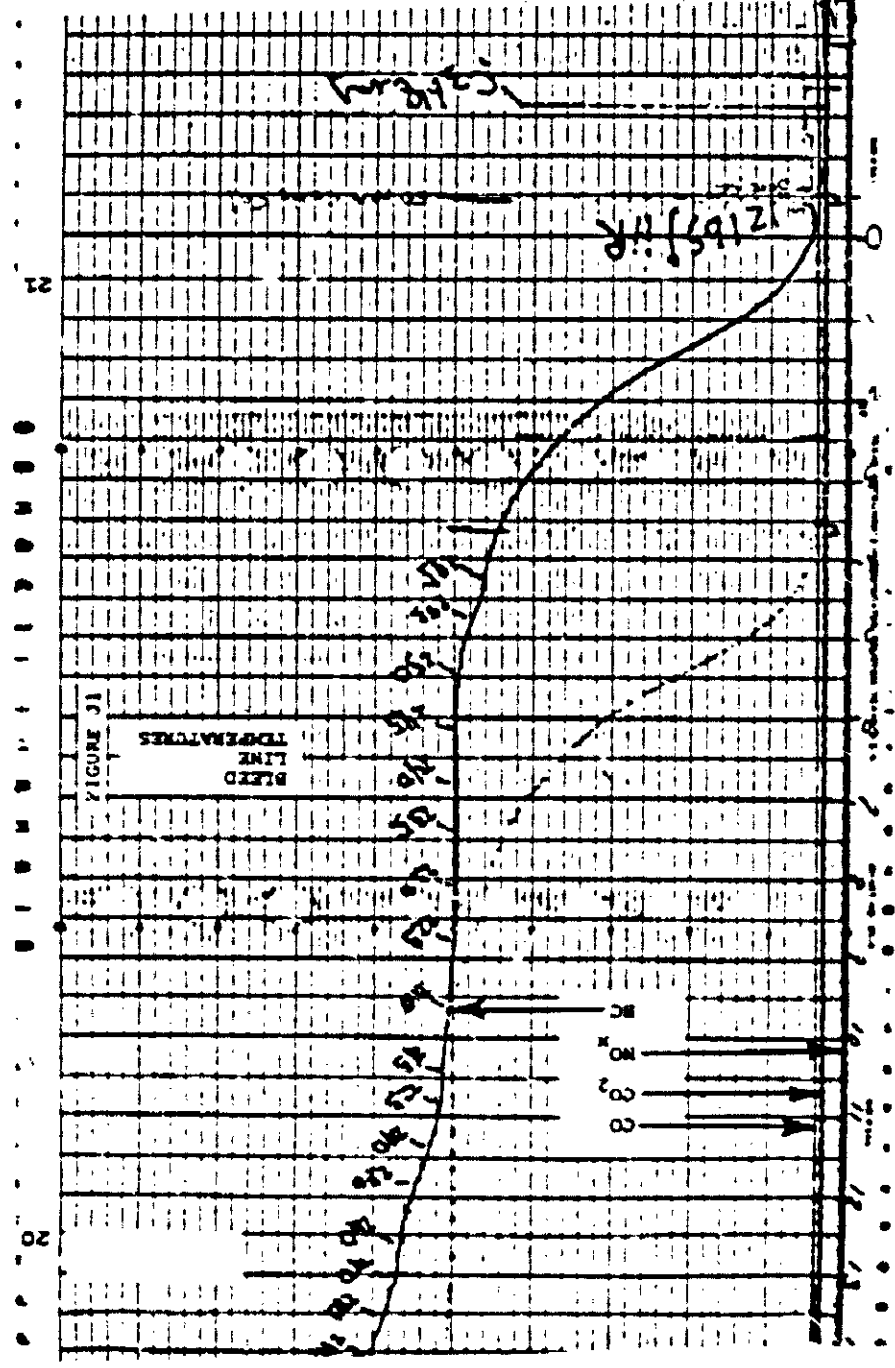
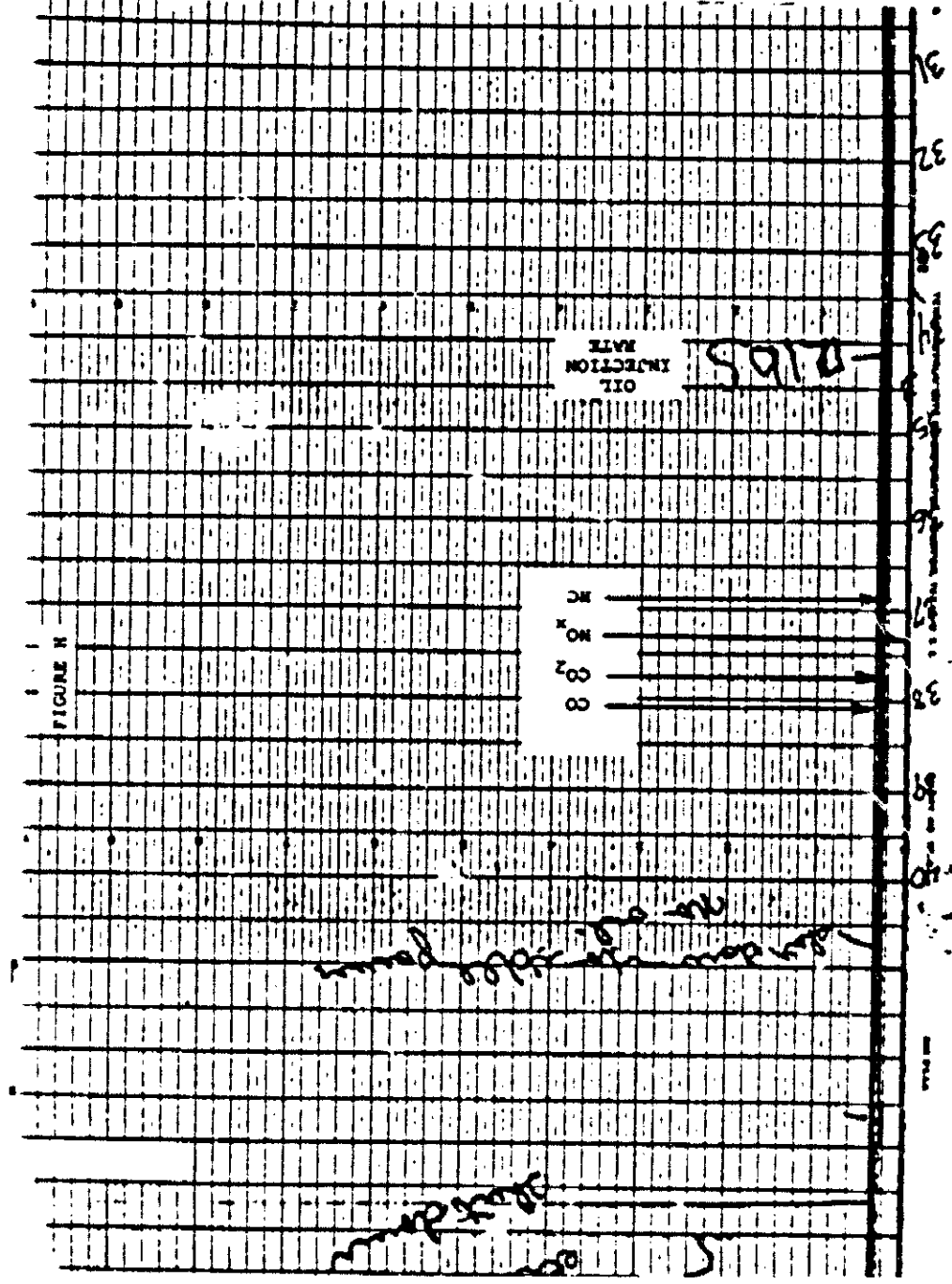
APPENDIX H

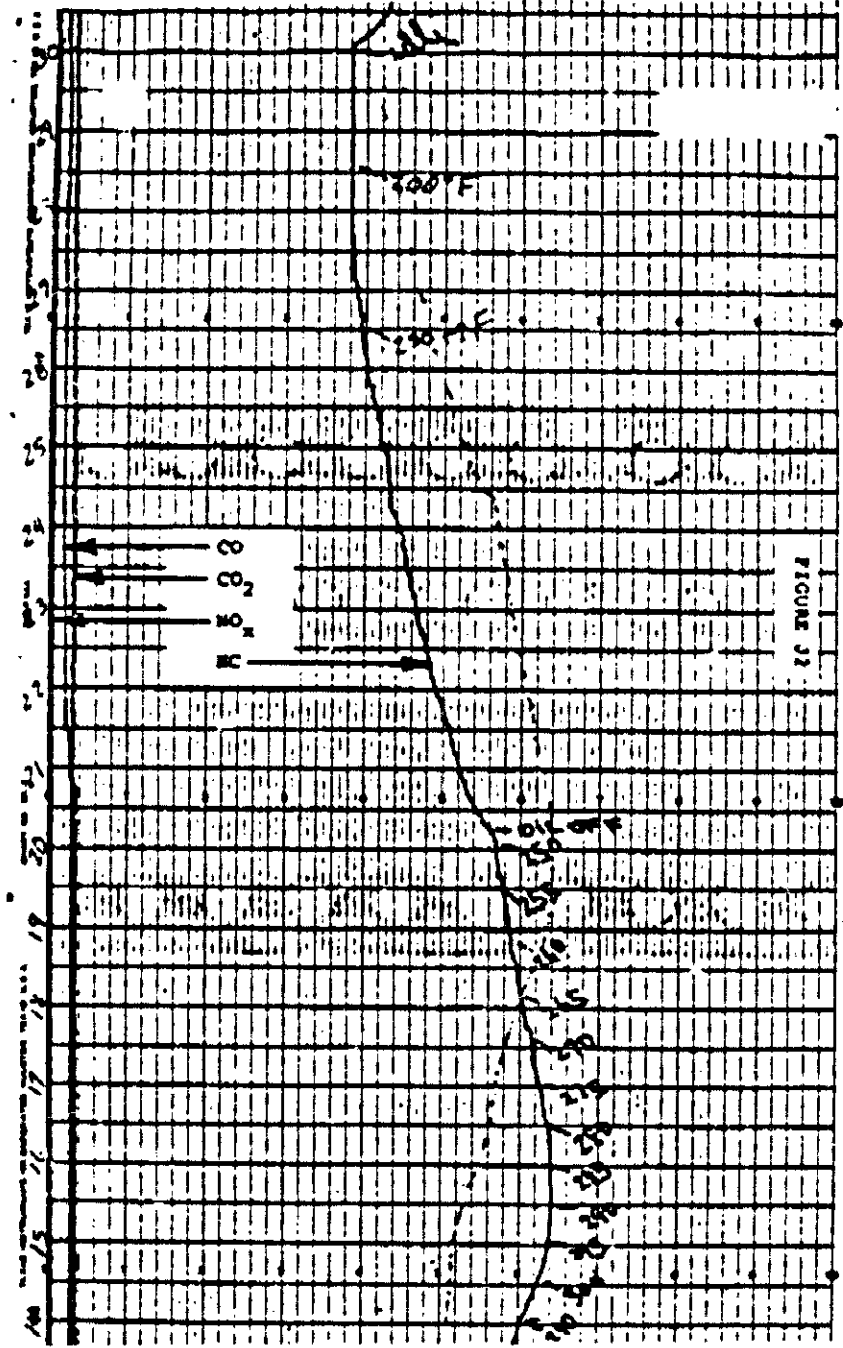
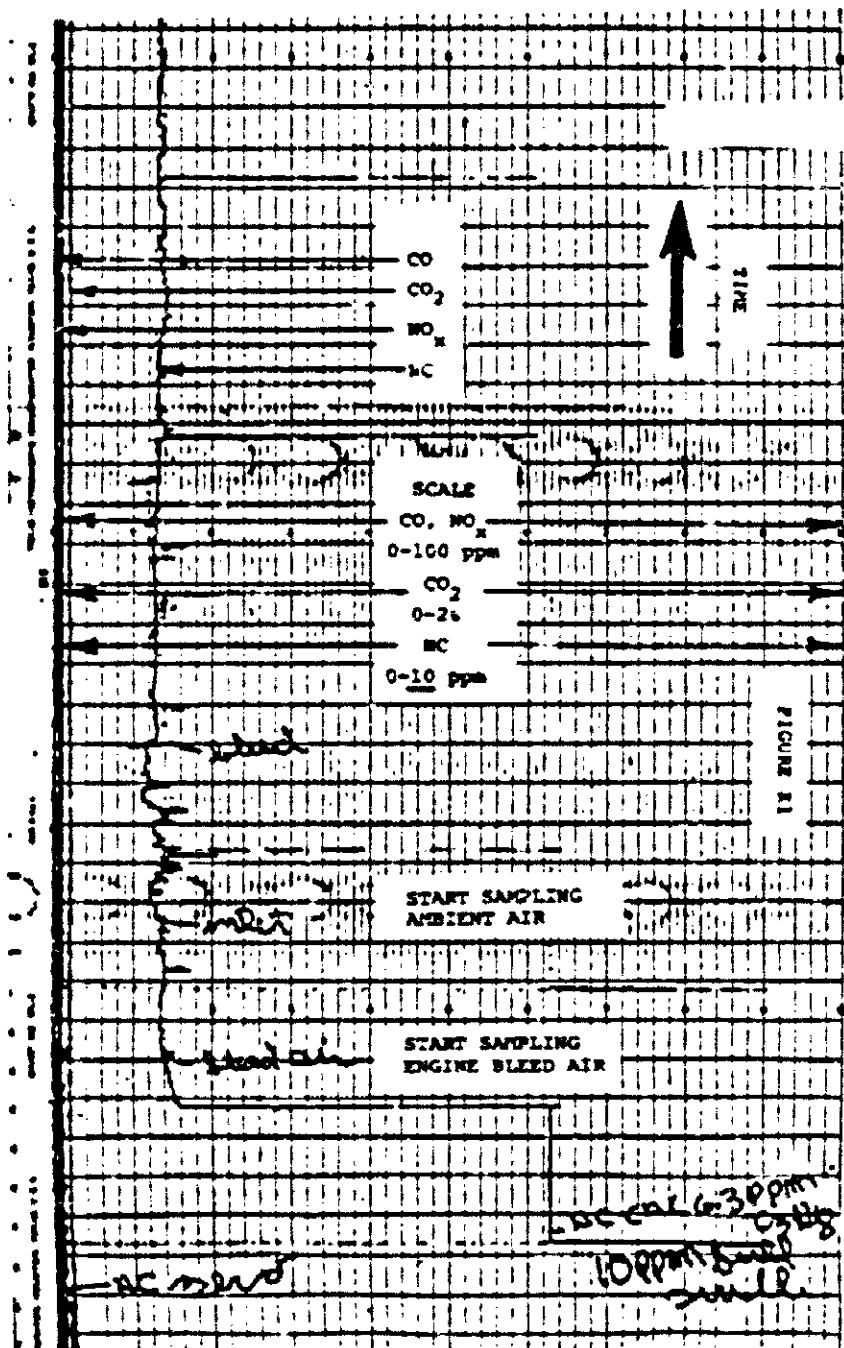
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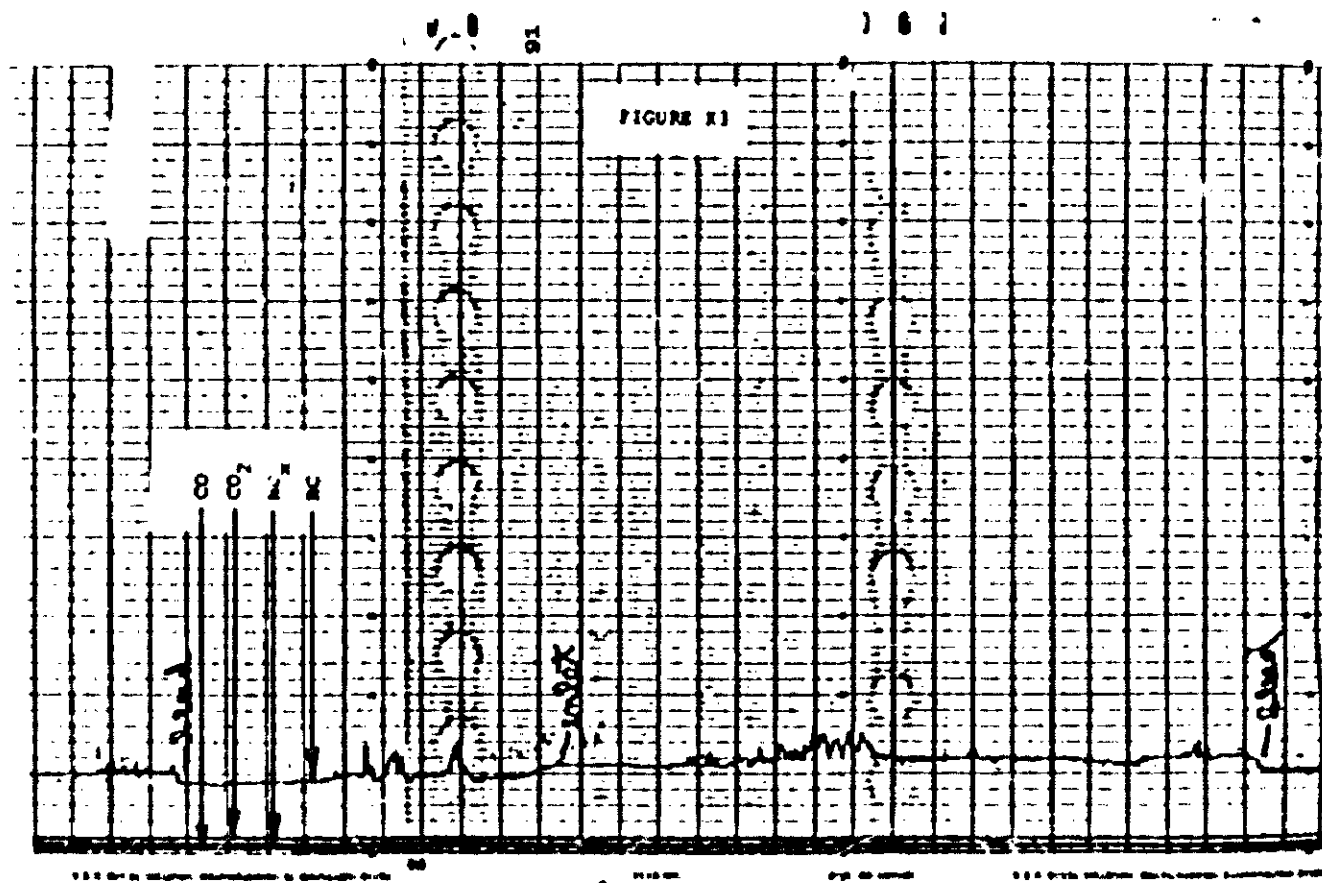
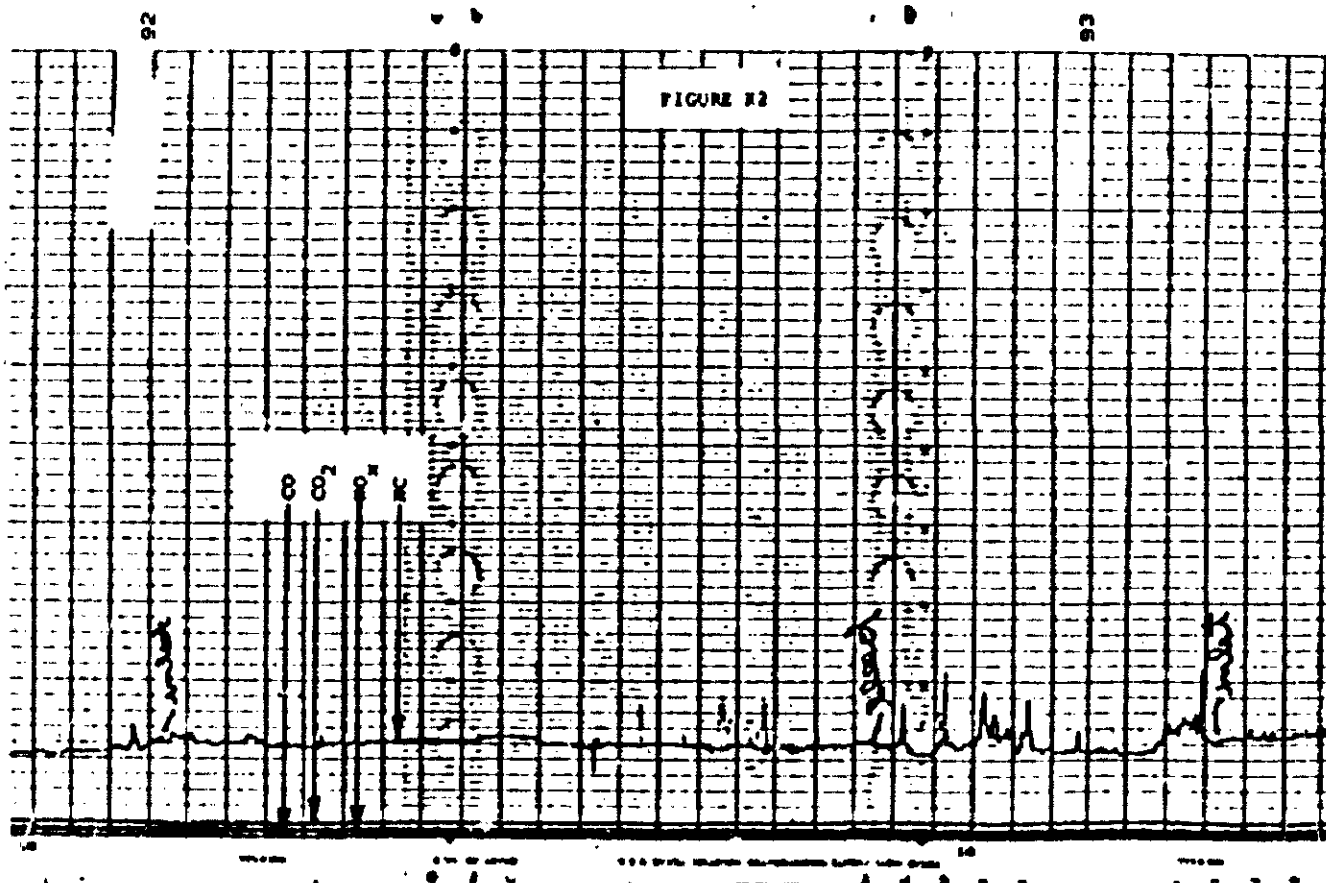


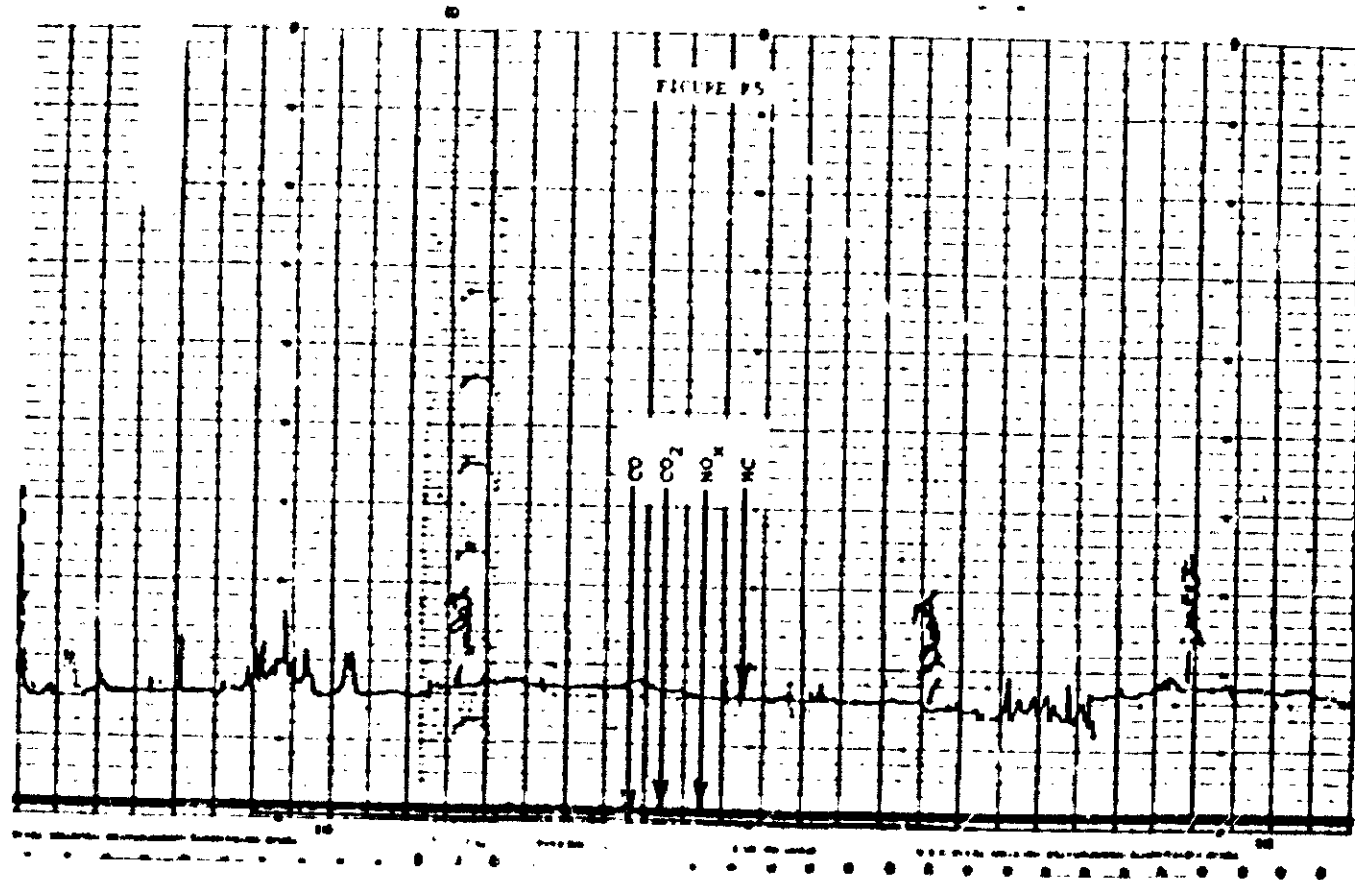
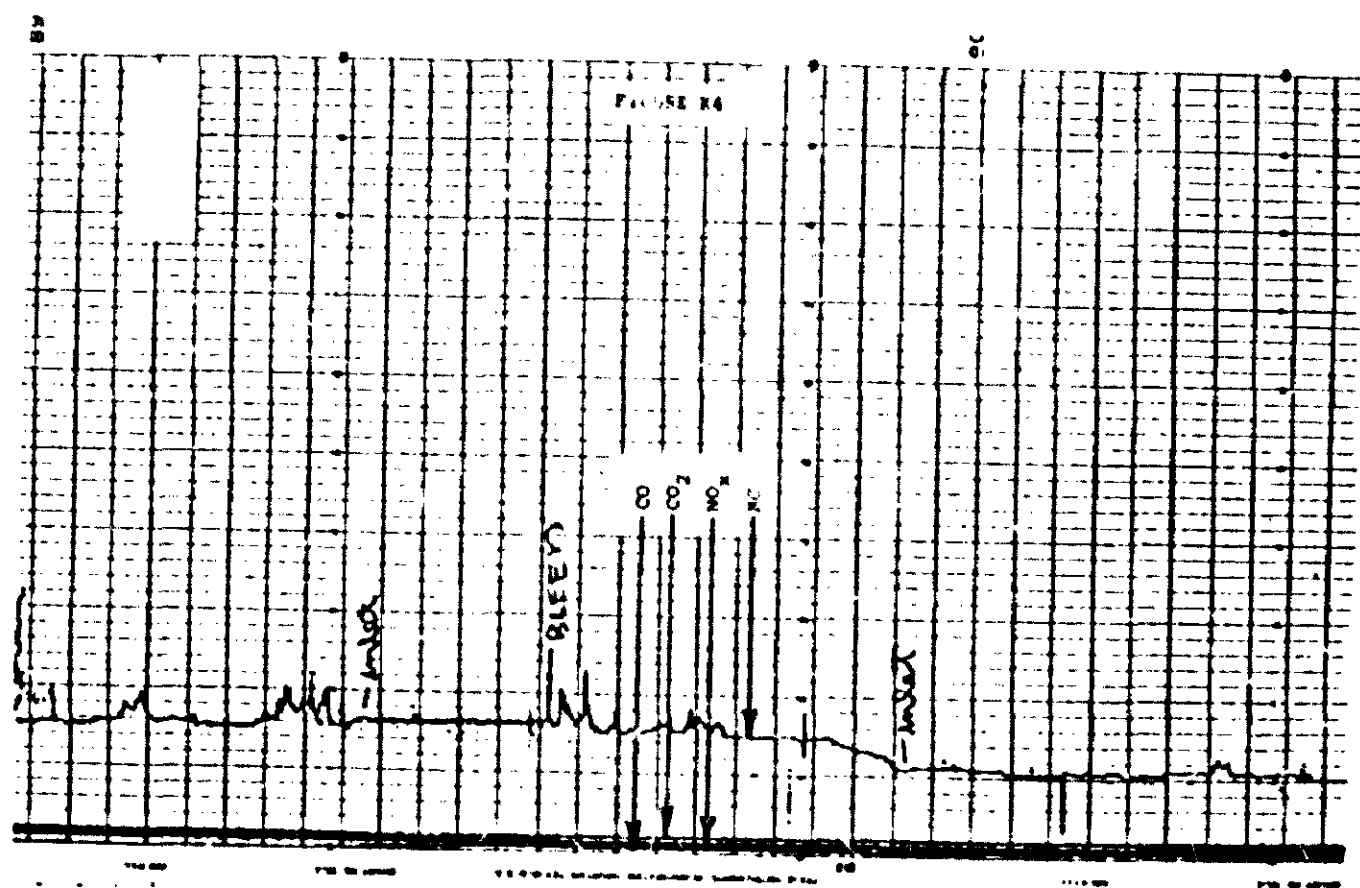


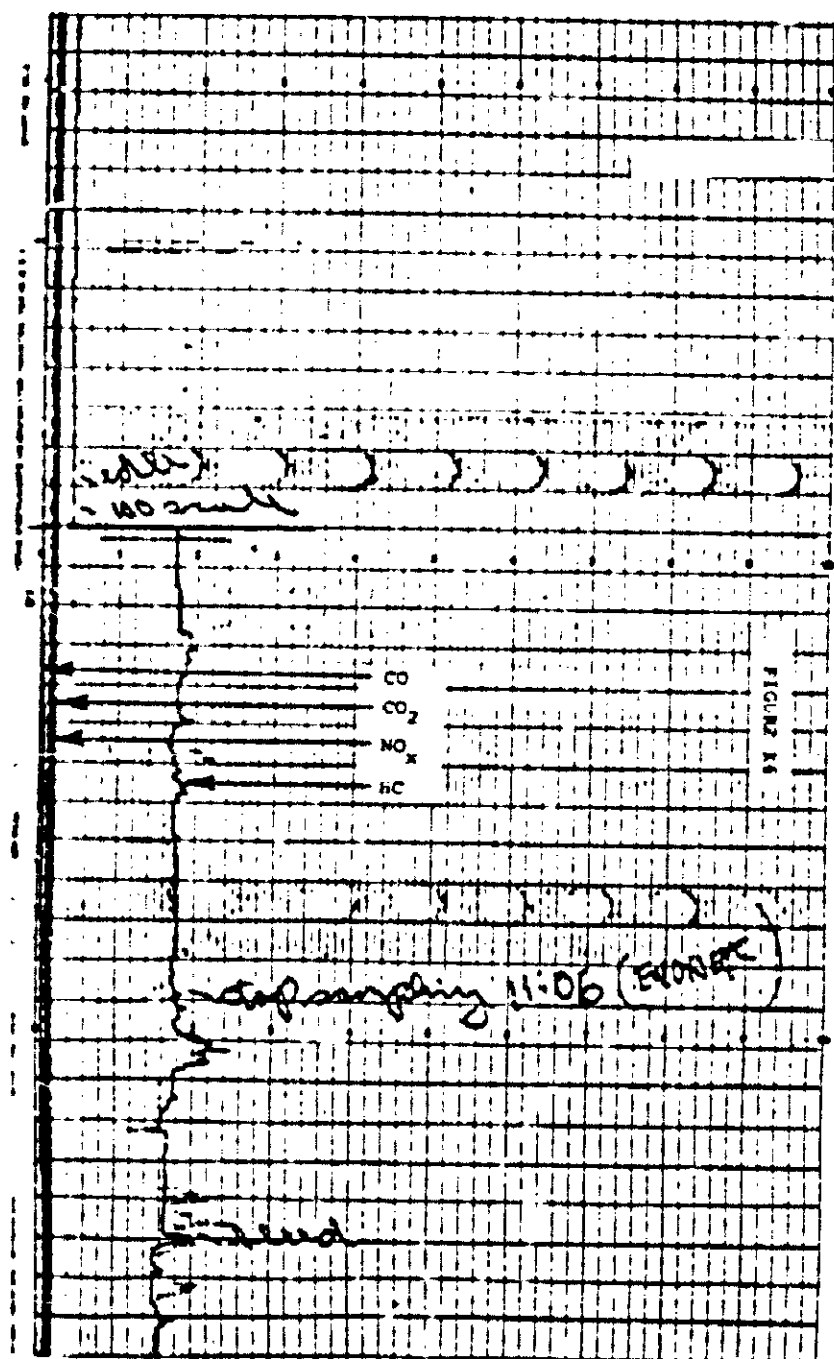
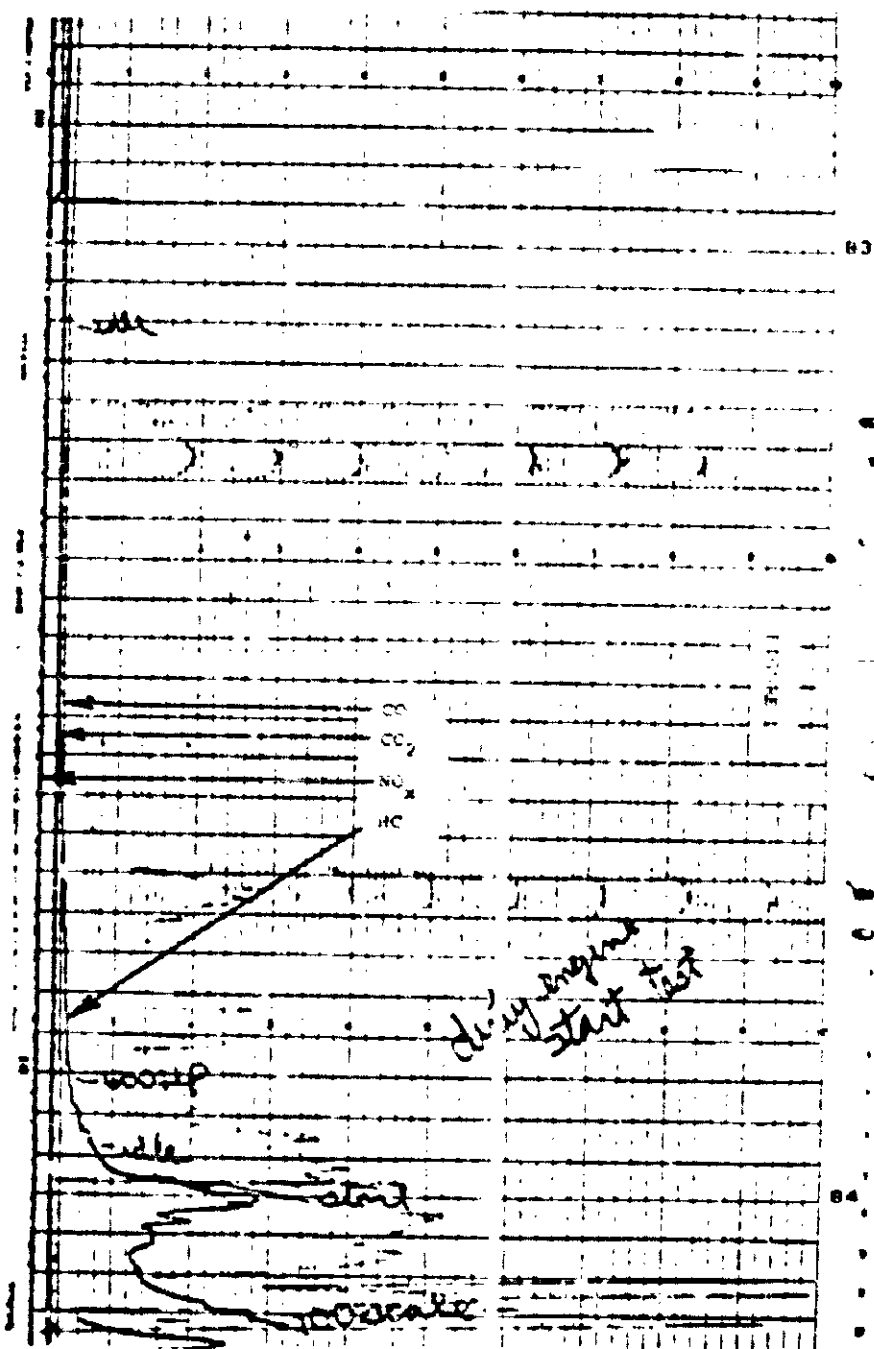












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